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The Crystal Structure of Tetraphenylphosphonium Trichloro-(*cis*-but-2-en-1,4-diol)platinum(II)

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Tetraphenylphosphonium trichloro(*cis*-but-2-en-1,4-diol)platinum(II), $(C_6H_5)_4P^+.[PtCl_3(C_4H_8O_2)]^-$, forms yellow monoclinic crystals, $a=13{\cdot}665$, $b=7{\cdot}471$, $c=27{\cdot}402$ Å, $\beta=102{\cdot}00°$, space group $P2_1/c$, with four formula units in the unit cell. The structure has been solved by standard methods and refined by the block-diagonal matrix least-squares methods, using 1899 photographic observations, to a final R value of 0·085. The $[PtCl_3(C_4H_8O_2)]^-$ anion is a π-complex and the platinum atom is square coordinated with the three chlorine atoms and the double bond of the olefinic diol. Two centrosymmetrically related anions interact through two O–H···O hydrogen bonds. The $(C_6H_5)_4P^+$ cation has almost $\bar{4}$ point symmetry. Intramolecular bond distances are normal.

During the investigation of the thermodynamics of the formation of platinum(II)-π-complexes with functionally substituted olefins, the X-ray crystal structure determination of some of them was undertaken. The purpose was to determine the mode of bonding of the substituted olefins and the effect of the substituents on the bonding to the metal.

Papers dealing with the structure of π-complexes of platinum(II) with unsaturated amines have been published by Spagna, Venanzi & Zambonelli (1970*a*, *b*).

In this article, the structure of tetraphenylphosphonium trichloro(*cis*-but-2-en-1,4-diol)platinum(II) is reported.

Experimental

Preparation

The compound $(C_6H_5)_4P^+.[PtCl_3(C_4H_8O_2)]^-$ was synthesized in the following manner. Commercial *cis*-but-2-en-1,4-diol (b.p. 102–107°/2·5–3 mmHg; dibenzoate, m.p. 65–66 °C from ethanol) (0·5 g) in 2*N*-hydrochloric acid (7–8 cm^3) was added to potassium tetrachloroplatinate (1 g) in 2*N*-hydrochloric acid (7–8 cm^3). The solution was heated (70 °C) until it became yellow, then cooled and filtered. The stoichiometric amount of tetraphenylphosphonium chloride was added to the solution, and the compound precipitated as a light yellow powder. Transparent yellow crystals suitable for single-crystal X-ray analysis were grown from dichloromethane–benzene solutions; m.p. 173–174 °C (found Pt, 26·7 %; $C_{28}H_{28}Cl_3O_2PPt$ requires Pt, 26·8 %).

Crystal data

Crystals of $(C_6H_5)_4P^+.[PtCl_3(C_4H_8O_2)]^-$ are stable in air and to X-rays. Preliminary X-ray and optical examinations of the crystals indicated that they belong to the monoclinic system. The space group, $P2_1/c$ (No. 14), was assigned on the basis of the systematic absences ($h0l$, $l=2n+1$; $0k0$, $k=2n+1$). Unit-cell dimensions at about 23 °C are: $a=13{\cdot}665\pm0{\cdot}002$, $b=7{\cdot}471\pm0{\cdot}001$, $c=27{\cdot}402\pm0{\cdot}003$ Å, $\beta=102{\cdot}00\pm0{\cdot}02$; these were determined by a least-squares refinement of seventy-six 2θ values measured on $0kl$ and $h0l$ Weissenberg photographs calibrated with superimposed ZnO powder lines, using Cu $K\alpha$ radiation ($\lambda=1{\cdot}5418$ Å).

The measured density (by flotation in a $ZnCl_2$ solution) of $1{\cdot}77\pm0{\cdot}01$ g.cm^{-3} agrees very well with the value of 1·769 g.cm^{-3} calculated for 4 units $(C_6H_5)_4P.[PtCl_3(C_4H_8O_2)]$ of formula weight 728·95 in the cell volume of 2736·26 Å^3.

The X-ray intensities were estimated visually from sets of multiple-film equi-inclination Weissenberg photographs taken, at room-temperature, about the a (9 layers, 0–8*kl*) and *b* (7 layers *h*0–6*l*) axes, with Ni-filtered Cu $K\alpha$ radiation. The intensities were corrected for Lorentz and polarization effects, and for spot extension (Phillips, 1954). Absorption corrections were applied as for cylindrical specimens [μ(Cu $K\alpha$) = 133·5 cm^{-1}] assuming approximate crystal radii of 0·05 mm for prisms approximately 0·1 × 0·2 × 0·3 mm in size.

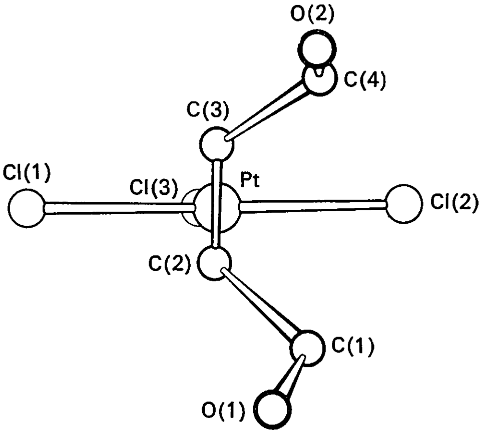

Fig. 1. The anion [$PtCl_3(C_4H_8O_2)$]$^-$ viewed along the Pt–olefin axis.

The two sets of data (about the *a* and *b* axes) were correlated by the method of Hamilton, Rollett & Sparks (1965). 1899 independent non-zero reflexions were collected and used in the analysis.

Structure determination

The structure was solved by the usual combination of Patterson and Fourier syntheses. Refinement was carried out by the block-diagonal matrix, (4 × 4) and (9 × 9), least-squares method, the quantity minimized being $w(|F_o|-|F_c|)^2$. The weighting scheme used, $w=(a+F_o+bF_o^2+cF_o^3)^{-1}$, was checked at intervals, and *a*, *b* and *c* adjusted, by means of a least-squares procedure, to keep a relatively constant average $\langle w|\Delta F|^2\rangle$ in groups of F_o or $\sin\theta/\lambda$ (Cruickshank, 1965). For the last cycles of the refinement, values of $a=24{\cdot}81$, $b=-0{\cdot}0187$ and $c=0{\cdot}00033$ were used. The standard deviation of an observation of unit weight was 0·7.

Table 1. *Final positional and thermal parameters*

Estimated standard deviations are given in parentheses.

The coefficients b_{ij} are defined by the expression $T=\exp\,[-10^{-4}(b_{11}h^2+b_{22}k^2+b_{33}l^2+b_{12}hk+b_{13}hl+b_{23}kl)]$.

Anion

	x	*y*	*z*	b_{11}	b_{12}	b_{13}	b_{22}	b_{23}	b_{33}
Pt	0·20899 (8)	0·20775 (15)	0·12921 (4)	65 (1)	−27 (3)	−3 (0)	163 (2)	−4 (1)	13 (0)
Cl(1)	0·3712 (6)	0·2111 (13)	0·1191 (3)	100 (6)	−15 (22)	39 (5)	305 (18)	−19 (11)	34 (2)
Cl(2)	0·0468 (6)	0·1901 (13)	0·1401 (3)	65 (5)	−79 (20)	28 (5)	398 (26)	46 (11)	33 (2)
Cl(3)	0·2512 (7)	−0·0234 (12)	0·1848 (3)	125 (7)	102 (21)	9 (5)	322 (22)	57 (9)	23 (1)
O(1)	0·0799 (19)	0·3284 (41)	−0·0216 (7)	144 (22)	168 (75)	11 (14)	611 (104)	−31 (29)	18 (3)
O(2)	0·1023 (15)	0·7484 (27)	0·0879 (7)	103 (16)	41 (45)	15 (12)	252 (57)	−1 (20)	25 (4)
C(1)	0·0741 (23)	0·2794 (54)	0·0280 (10)	86 (23)	−79 (90)	1 (16)	525 (116)	25 (37)	16 (4)
C(2)	0·1749 (22)	0·3406 (34)	0·0604 (7)	131 (27)	−12 (61)	2 (14)	187 (61)	−1 (20)	9 (3)
C(3)	0·1933 (23)	0·4749 (41)	0·0966 (7)	146 (29)	63 (77)	30 (14)	302 (76)	13 (23)	7 (3)
C(4)	0·1129 (14)	0·5694 (38)	0·1161 (9)	7 (13)	−18 (48)	0 (11)	273 (68)	9 (29)	23 (4)

Cation

	x	*y*	*z*	b_{11}	b_{12}	b_{13}	b_{22}	b_{23}	b_{33}
P	−0·3017 (5)	0·3875 (9)	0·1393 (2)	52 (4)	−19 (14)	10 (3)	195 (13)	−8 (5)	11 (1)
C(5)	−0·1967 (15)	0·5398 (31)	0·1572 (7)	35 (14)	−40 (45)	7 (10)	161 (47)	−21 (19)	13 (3)
C(6)	−0·1830 (20)	0·6735 (36)	0·1219 (9)	68 (19)	35 (56)	34 (15)	224 (70)	−6 (26)	20 (4)
C(7)	−0·1006 (20)	0·7882 (42)	0·1367 (9)	78 (20)	−4 (68)	14 (15)	237 (58)	−16 (30)	19 (4)
C(8)	−0·0359 (19)	0·7857 (43)	0·1856 (10)	62 (19)	248 (73)	37 (16)	333 (74)	44 (34)	24 (5)
C(9)	−0·0571 (20)	0·6414 (42)	0·2172 (9)	74 (21)	−26 (64)	30 (15)	276 (67)	−54 (29)	19 (4)
C(10)	−0·1373 (19)	0·5184 (35)	0·2056 (8)	83 (20)	−65 (58)	−9 (13)	191 (56)	−29 (24)	14 (3)
C(11)	−0·4054 (14)	0·5319 (34)	0·1207 (8)	12 (11)	25 (47)	3 (10)	231 (57)	31 (23)	17 (3)
C(12)	−0·4223 (16)	0·6605 (32)	0·1559 (9)	26 (14)	−67 (46)	−1 (12)	199 (60)	−48 (24)	21 (4)
C(13)	−0·5104 (20)	0·7717 (39)	0·1423 (11)	65 (18)	−59 (59)	13 (15)	200 (59)	9 (29)	24 (5)
C(14)	−0·5753 (19)	0·7632 (44)	0·0969 (10)	55 (18)	−32 (66)	30 (15)	374 (91)	67 (32)	21 (5)
C(15)	−0·5527 (18)	0·6263 (46)	0·0626 (8)	50 (17)	−92 (67)	26 (12)	440 (87)	8 (29)	12 (3)
C(16)	−0·4731 (16)	0·5149 (32)	0·0763 (8)	35 (14)	87 (48)	6 (11)	186 (51)	19 (22)	15 (3)
C(17)	−0·2908 (18)	0·2404 (29)	0·0876 (6)	80 (18)	65 (47)	−10 (10)	178 (59)	−9 (16)	5 (2)
C(18)	−0·3567 (21)	0·1047 (42)	0·0750 (8)	89 (22)	−95 (68)	−17 (13)	276 (72)	−27 (27)	14 (3)
C(19)	−0·3515 (23)	−0·0008 (36)	0·0356 (10)	115 (26)	17 (65)	−20 (17)	149 (53)	−9 (28)	22 (5)
C(20)	−0·2797 (22)	0·0354 (46)	0·0070 (10)	89 (23)	219 (78)	−16 (16)	370 (89)	−40 (32)	20 (5)
C(21)	−0·2135 (21)	0·1713 (57)	0·0175 (11)	53 (19)	193 (88)	12 (16)	662 (154)	−13 (44)	21 (5)
C(22)	−0·2109 (18)	0·2863 (45)	0·0613 (10)	42 (15)	91 (68)	11 (13)	384 (82)	−61 (34)	21 (4)
C(23)	−0·3145 (16)	0·2456 (29)	0·1895 (8)	44 (15)	−56 (43)	7 (12)	157 (59)	−2 (19)	16 (3)
C(24)	−0·2389 (19)	0·1123 (35)	0·2060 (9)	76 (20)	8 (55)	−19 (13)	155 (52)	44 (24)	18 (4)
C(25)	−0·2491 (21)	−0·0006 (40)	0·2427 (9)	88 (22)	74 (67)	32 (16)	244 (67)	−5 (28)	19 (4)
C(26)	−0·3347 (22)	−0·0004 (35)	0·2613 (9)	106 (24)	−30 (62)	−8 (16)	150 (54)	15 (25)	19 (4)
C(27)	−0·4148 (22)	0·1177 (45)	0·2445 (9)	99 (24)	−3 (75)	24 (16)	297 (75)	−13 (31)	17 (4)
C(28)	−0·4024 (22)	0·2490 (30)	0·2071 (9)	106 (23)	−39 (53)	28 (15)	146 (67)	−1 (20)	16 (4)

The initial refinement, with individual isotropic temperature factors assigned to all atoms, converged to an *R* value of 0·115. A further refinement was performed in which all atoms were allowed to vibrate anisotropically: this converged to $R=0·085$. The parameter shifts in the final cycle of refinement were all less than one fifth of their estimated standard deviations.

No attempt was made to include the contribution of the hydrogen atoms. No region of the electronic density exceeded $\pm 3\sigma(\varrho)$ on the final difference Fourier synthesis [$\sigma(\varrho)=0·41$ e.Å^{-3}]. Table 1 gives the final positional and anisotropic thermal parameters for the non-hydrogen atoms. The magnitudes and orientation of the thermal vibrational ellipsoids are reported in

Table 2. *Magnitudes and orientation of thermal ellipsoids*

Anion

	r.m.s. amplitudes	Angles with respect to				r.m.s. amplitudes	Angles with respect to		
		a	*b*	*c*			*a*	*b*	*c*
Pt	0·194 Å	63°	58°	54°	O(2)	0·259 Å	69°	159°	91°
	0·220	84	145	58		0·295	69	79	35
	0·268	152	79	53		0·320	150	108	55
Cl(1)	0·287	153	69	62	C(1)	0·233	70	93	32
	0·290	66	25	88		0·281	153	110	61
	0·357	77	103	29		0·398	108	21	77
Cl(2)	0·213	156	113	72	C(2)	0·179	87	89	15
	0·317	70	129	52		0·229	92	177	88
	0·380	101	47	43		0·353	176	88	75
Cl(3)	0·228	73	131	52	C(3)	0·140	102	93	3
	0·335	120	60	38		0·284	73	163	93
	0·361	36	56	90		0·371	21	73	89
O(1)	0·254	94	80	13	C(4)	0·077	13	86	90
	0·323	34	122	92		0·274	92	156	66
	0·454	124	146	77		0·300	103	66	24

Cation

	r.m.s. amplitudes	Angles with respect to				r.m.s. amplitudes	Angles with respect to		
		a	*b*	*c*			*a*	*b*	*c*
P	0·199 Å	87°	75°	22°	C(17)	0·124 Å	79°	88°	23°
	0·214	158	110	70		0·205	114	24	83
	0·241	68	154	82		0·303	153	114	69
C(5)	0·160	39	59	79	C(18)	0·165	63	66	47
	0·206	128	60	44		0·284	104	143	54
	0·241	83	134	48		0·335	149	64	64
C(6)	0·201	143	67	53	C(19)	0·204	92	5	85
	0·260	105	155	68		0·243	60	94	42
	0·286	57	81	46		0·376	150	94	48
C(7)	0·245	86	41	51	C(20)	0·188	41	125	81
	0·266	22	107	88		0·248	91	63	29
	0·278	111	126	39		0·416	131	133	63
C(8)	0·087	146	56	80	C(21)	0·178	19	108	97
	0·275	104	110	20		0·279	93	85	10
	0·384	59	41	73		0·453	108	161	83
C(9)	0·204	117	58	37	C(22)	0·165	156	67	72
	0·252	30	53	89		0·248	74	67	37
	0·322	77	136	53		0·369	107	147	59
C(10)	0·169	67	53	53	C(23)	0·166	42	49	90
	0·260	86	141	53		0·235	62	133	63
	0·308	157	79	58		0·249	119	70	27
C(11)	0·102	15	97	89	C(24)	0·158	72	134	56
	0·223	104	131	42		0·237	53	48	72
	0·281	94	42	48		0·331	138	76	40
C(12)	0·115	37	63	77	C(25)	0·213	131	53	54
	0·232	126	43	62		0·268	90	128	39
	0·305	97	121	31		0·309	41	59	77
C(13)	0·205	48	42	97	C(26)	0·200	94	168	78
	0·267	48	128	74		0·245	63	82	40
	0·302	109	75	17		0·346	153	81	52
C(14)	0·172	143	112	51	C(27)	0·240	107	76	15
	0·260	54	113	55		0·292	110	158	78
	0·359	95	32	58		0·301	27	106	81
C(15)	0·145	142	102	43	C(28)	0·198	103	165	79
	0·234	56	81	47		0·231	106	78	13
	0·362	75	165	93		0·314	21	99	83
C(16)	0·132	37	122	84					
	0·237	116	103	19					
	0·260	66	35	71					

Table 2. The final values of observed and calculated structure factors (in electrons ×10) for the reflexions used in the analysis are presented in Table 3.

Atomic scattering curves given by Cromer & Mann (1968) were used, all atoms being used as neutral species. The effects of anomalous dispersion were included

Table 3. *Observed and calculated structure factors* (*in electrons* × 10) $F(000)=1408\cdot72$

K	L	|FO|	FC
•••• H = 0 ••••			
0	4	2281	-4558
0	6	415	532
0	8	2180	2448
0	10	475	-527
0	12	2002	-2208
0	14	846	1002
0	16	1713	1776
0	18	1426	-1206
0	20	1033	-903
0	22	611	603
0	24	531	512
0	26	633	-608
0	28	393	-370
0	30	345	328
0	32	315	284
0	34	185	-175
1	2	3227	-3326
1	3	212	-230
1	5	747	668
1	6	1479	2467
1	7	558	514
1	8	515	-502
1	10	1771	-2408
1	11	173	128
1	12	736	674
1	13	518	-542
1	14	1756	1920
1	15	326	358
1	16	841	-860
1	17	258	246
1	18	1182	-1173
1	19	575	-530
1	20	621	641
1	22	712	703
1	23	304	273
1	24	447	-478
1	26	560	-498
1	28	517	442
1	30	266	259
1	32	288	-240
1	34	121	-134
2	0	2584	-2863
2	1	1701	-1703
2	2	547	-501
2	3	458	-441
2	4	3045	3147
2	5	765	695
2	6	383	-329
2	7	996	955
2	8	1991	-2098
2	9	978	-1040
2	10	397	422
2	11	675	-753
2	12	1539	1771
2	13	616	640
2	14	719	-826
2	16	893	-913
2	17	627	-620
2	18	607	565
2	19	342	-341
2	20	857	813
2	21	321	313
2	22	461	-428
2	23	378	349
2	24	506	-469
2	25	405	-347
2	26	532	443
2	28	453	403
2	29	302	236
2	30	226	-219
2	32	249	-216
2	33	186	-145
3	1	1072	-1021
3	2	1754	1684
3	3	1893	1008
3	4	374	-335
3	5	1608	1546
3	6	1149	-1067
3	7	1358	-1258
3	8	258	238
3	9	1120	-1148
3	10	1169	1132
3	11	1081	1067
3	13	267	324
3	14	996	-1063
3	15	752	-604
3	18	732	774
3	19	598	630
3	22	588	-567
3	23	805	-547
3	25	166	-146
3	26	341	303
3	27	475	431
3	30	341	-321
3	31	248	-230
4	0	1635	1660
4	1	1553	1546
4	3	1053	1074
4	4	960	-869
4	5	1589	-1546
4	7	864	-852
4	8	516	489
4	9	1452	1334
4	10	353	352
4	11	619	660
4	12	401	-415
4	13	905	-925
4	14	208	-205
4	15	299	-335
4	16	392	448
4	17	792	803
4	18	218	-265
4	20	281	-309
4	21	707	-701
4	24	264	295
4	25	579	509
4	28	259	-245
4	29	327	-293
5	1	1176	1196
5	3	1514	-1425
5	5	867	-832
5	7	1581	1492
5	9	655	603
5	10	365	-329
5	11	1085	-1134
5	13	466	-543
5	15	833	945
5	17	217	211
5	19	729	-620
5	23	662	555
5	27	328	-298
6	1	849	-826
6	3	851	-900
6	5	1064	1049
6	7	691	706
6	9	988	-1003
6	11	436	-446
6	13	765	838
6	17	574	-634
6	21	442	451
6	25	383	-348
7	1	608	-634
7	3	744	717
7	5	516	531
7	7	648	-602
7	9	380	-371
7	11	526	568
7	15	517	-497
7	19	393	358
7	23	310	-301
8	1	327	301
8	4	372	354
8	5	312	-204
8	7	253	-250
8	8	351	-314
8	11	196	209
8	12	213	216
8	13	217	-245
8	15	230	-216
8	16	201	-191
8	17	281	209
9	1	190	233
9	2	352	319
9	3	187	-215
9	6	276	-204
9	10	198	212
9	11	125	-106
•••• H = 1 ••••			
0	4	414	-415
0	-4	363	-359
0	6	3966	3822
0	-6	1762	-2019
0	8	560	600
0	-8	554	527
0	10	1531	-1424
0	-10	1311	1437
0	12	698	-638
0	-12	674	-702
0	14	1739	1919
0	-14	1381	-1924
0	18	1402	-1505
0	-16	941	1034
0	-20	800	-838
0	22	948	915
0	-22	510	-530
0	24	347	-383
0	-24	528	544
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1	-3	401	-381
1	-4	670	-684
1	5	470	-588
1	6	644	662
1	-8	706	688
1	9	586	427
1	-9	411	-406
1	10	786	-810
1	-10	416	348
1	-12	667	-535
1	14	750	695
1	-20	523	-527
2	0	439	-470
2	-1	461	455
2	2	470	-581
2	-2	324	408
2	3	613	-638
2	4	619	698
2	-4	323	342
2	-5	355	-348
2	6	379	488
2	-6	563	-592
2	7	350	395
2	8	523	-657
2	-9	331	314
2	-10	386	365
2	-12	341	312
2	-14	475	-535
2	15	346	451
2	16	338	-406
2	-16	354	-359
2	-18	358	444
3	1	613	-745
3	-3	529	503
3	-4	427	459
3	5	487	518
3	6	365	-483
3	-6	748	-803
3	9	452	-447
3	10	481	614
3	-11	440	455
3	-12	486	533
3	14	459	-567
4	-5	563	590
•••• H = 10 ••••			
0	0	903	991
0	2	361	-317
0	-2	564	533
0	4	1202	-1237
0	-4	1075	-965
0	6	744	715
0	-6	508	-412
0	8	908	1024
0	-8	1122	991
0	10	628	-614
0	12	485	-484
0	-12	815	-858
0	14	563	516
0	-16	556	574
0	18	524	-550
0	-20	507	-461
0	22	413	479
0	-24	463	438
1	2	620	-563
1	-2	882	852
1	4	661	575
1	-6	606	-631
1	-10	818	778
1	-14	781	-823
1	-16	740	639
1	-22	544	-446
2	0	743	-756
2	2	325	341
2	4	529	380
2	-4	500	456
2	5	355	390
2	6	332	-401
2	-6	396	425
2	-7	344	-334
2	8	335	-351
2	-8	545	-369
2	-10	324	-287
2	-11	434	466
2	-12	436	523
2	-15	530	-515
2	-16	547	-576
2	-19	289	335
2	-20	405	441
2	-24	366	-396
3	-1	576	-521
3	-2	514	-496
•••• H = 11 ••••			
0	0	487	-482
0	-2	940	877
0	6	581	560
0	-6	1075	-1074
0	-8	438	-319
0	10	541	-611
0	-10	949	968
0	-14	853	-722
0	-18	700	620
0	-22	681	-560
1	0	553	-557
1	2	455	433
1	4	663	605
1	-4	851	766
1	-8	866	-768
1	-12	736	776
1	-16	739	-778
1	-20	595	480
2	-1	503	-554
2	2	506	639
2	-2	648	-761
2	3	392	453
2	-3	476	404
2	6	357	-474
2	-6	791	671
2	7	355	-410
2	-11	413	330
2	-14	584	584
2	-15	357	-338
2	-16	494	-493
2	-22	452	398
3	0	452	482
3	1	521	536
3	-3	452	-517
3	4	491	-544
3	-4	522	-688
3	5	415	-472
3	-7	421	435
3	8	499	572
3	-8	560	619
3	-11	368	-415
3	-12	450	-523
•••• H = 12 ••••			
0	0	1060	-923
0	4	1040	849
0	-4	1247	1011
0	8	561	-499
0	-8	1180	-969
0	-12	750	713
0	-14	423	-514
0	16	416	-435
0	-16	512	-446
0	20	193	285
1	-2	768	-808
1	6	931	-918
1	-6	842	761
1	10	691	624
1	-10	726	-675
2	0	582	683
2	-3	333	-308
2	4	453	-558
2	-4	531	-502
2	6	365	475
2	-8	546	559
2	-12	526	-519
2	-16	487	528
2	-20	337	-330
•••• H = 13 ••••			
0	2	701	627
0	-2	664	-576
0	6	653	-645
0	-6	745	631
0	-10	602	-506
0	-14	605	551
0	18	168	248
0	-16	471	-419
1	0	550	532
1	4	532	-466
1	-4	625	-594
1	-8	554	491
2	1	373	422
2	2	409	-455
2	-2	400	437
2	-3	368	-398
2	6	346	404
2	-6	348	-398
•••• H = 14 ••••			
0	0	379	338
0	4	489	-392

in the calculated structure factors (Ibers & Hamilton, 1964); the values of $\Delta f'$ for Pt, Cl and P were those computed by Cromer (1965), and the imaginary part of the anomalous dispersion factors was ignored.

Calculations were carried out on the UNIVAC 1108 computer of Rome University and were performed with the system of programs developed at the Laboratorio di Strutturistica Chimica 'Giordano Giacomello' (Domenicano, Spagna & Vaciago, 1969).

Results and discussion

Anion

The inner coordination geometry of the anion $[PtCl_3(C_4H_8O_2)]^-$ is shown in Figs. 1 and 2. If one considers the olefin as a monodentate ligand, the platinum atom is four-coordinate with the three chlorine atoms and the double bond of the but-2-ene-1,4-diol in an essentially square-planar configuration.

The olefinic double bond lies approximately perpendicular to the equatorial plane and is bisected by it (Fig. 1). The line C(2)–C(3) and the plane [Pt C(2) C(3)] make angles of 85 and 88° respectively, with the best plane through Pt, Cl(1), Cl(2), Cl(3) and the centre of the C(2)–C(3) bond ($0{\cdot}069x+0{\cdot}665y+0{\cdot}713z-3{\cdot}734=0$, in terms of monoclinic coordinates).

The C=C bond distance (1·40 (4) Å) shows the usual lengthening on attachment of the olefin to the metal.

These results are similar to those found in the anion in Zeise's salt (Black, Mais & Owston, 1969; Hamilton, 1969), and in other platinum(II)-olefin π-complexes (Alderman, Owston & Rowe, 1960; Mason & Robertson, 1969; Spagna *et al.*, 1970*a*, *b*).

The three Pt–Cl bonds are almost equivalent with an average length of 2·297 (5) Å. This value is in good agreement with the sum of the covalent radii (2·29 Å) and with the values found for Pt–Cl bonds in the *cis* position with respect to an olefin ligand (Spagna *et al.*, 1970*a*, *b*). In this case there does not appear to be any evidence for a weakening of the bond *trans* to the coordinating olefin.

The butenylendiol molecule maintains its *cis* configuration on complexing with platinum, although the methylenic groups bend away from the metal atom. An indication of the degree of the bending back of the methylenic groups is provided by the values of the dihedral angles between the [Pt C(2) C(3)] plane and the [C(1) C(2) C(3)] and [C(2) C(3) C(4)] planes: these are 107 and 97° respectively. Therefore the four carbon atoms are not planar, since a torsion angle of 10° is found about the C(2)–C(3) bond (Fig. 3).

An interesting feature of this compound is the solid state dimerization of two centrosymmetrically related

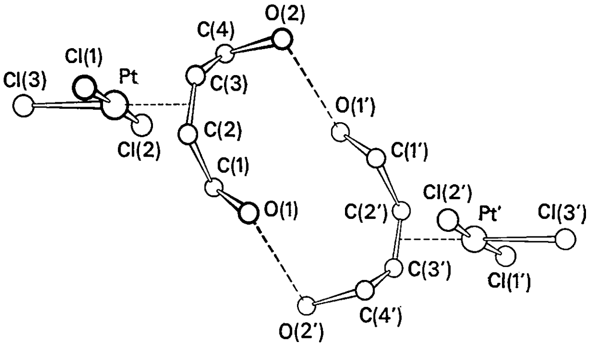

Fig. 2. Two centrosymmetrically related, hydrogen bonded, anions $[PtCl_3(C_4H_8O_2)]^-$.

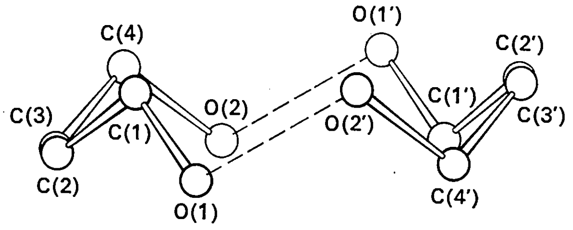

Fig. 3. The conformation of the but-2-ene-1,4-diol group. The two centrosymmetrically related molecules are hydrogen bonded.

Table 4. *Bond lengths and angles in the complex anion* $[PtCl_3(C_4H_8O_2)]^-$

Estimated standard deviations are given in parentheses.

MP defines the midpoint of the olefinic double bond; it was assigned standard deviations equal to those of C(2) and C(3). Although the olefinic carbon atoms C(2) and C(3) cannot be said to be bound to the platinum atom, their distances from the metal atom, as well as the angle C(2)–Pt–C(3), are quoted. The superscript primes refer to atoms belonging to the unit in position $-x, 1-y, -z$.

Pt—Cl(1)	2·291 (9) Å	Cl(1)–Pt—Cl(2)	177·3 (4)°
Pt—Cl(2)	2·301 (8)	Cl(1)–Pt—Cl(3)	88·4 (3)
Pt—Cl(3)	2·296 (9)	Cl(1)–Pt—MP	86.4 (9)
Pt—MP	2·02 (3)	Cl(2)–Pt—Cl(3)	89·2 (3)
Pt—C(2)	2·10 (2)	Cl(2)–Pt—MP	95·9 (9)
Pt—C(3)	2·18 (3)	Cl(3)–Pt—MP	174·8 (9)
O(1)–C(1)	1·43 (4)	C(2) –Pt—C(3)	38·1 (9)
C(1)–C(2)	1·54 (4)	O(1) –C(1)–C(2)	105 (3)
C(2)–C(3)	1·40 (4)	C(1) –C(2)–C(3)	129 (3)
C(3)–C(4)	1·50 (4)	C(2) –C(3)–C(4)	124 (3)
C(4)–O(2)	1·54 (3)	C(3) –C(4)–O(2)	103 (2)
O(1)···O(2')	2·82 (3)	C(1) –O(1)···O(2')	110 (2)
		O(1)···O(2')–C(4')	97 (1)

anions, through two O–H· · ·O hydrogen bonds [2·82 (3) Å in length], (Figs. 2 and 3). The existence of these hydrogen bonds probably stabilizes the conformation of the butenylendiol, which is quite unusual for a six-termed chain, with dihedral angles:

O(1)-[C(1)-C(2)]-C(3)	−113°
C(1)-[C(2)-C(3)]-C(4)	−10
C(2)-[C(3)-C(4)]-O(2)	101 .

Bond distances and angles related to the anion $[PtCl_3(C_4H_8O_2)]^-$ are reported in Table 4.

Cation

The cation $(C_6H_5)_4P^+$, viewed along the crystallographic *b* axis in Fig. 4, has almost $\bar{4}$ point symmetry. The parameters reported in Tables 5 and 6 give an indication of the degrees of the deviation from regular symmetry around the phosphorus atom.

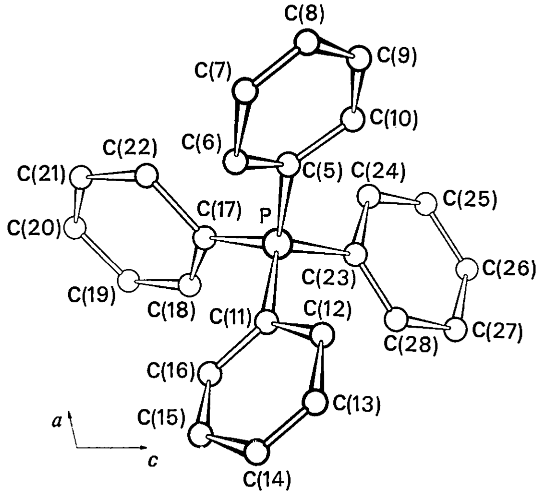

Fig. 4. The tetraphenylphosphonium cation viewed along the *b* axis.

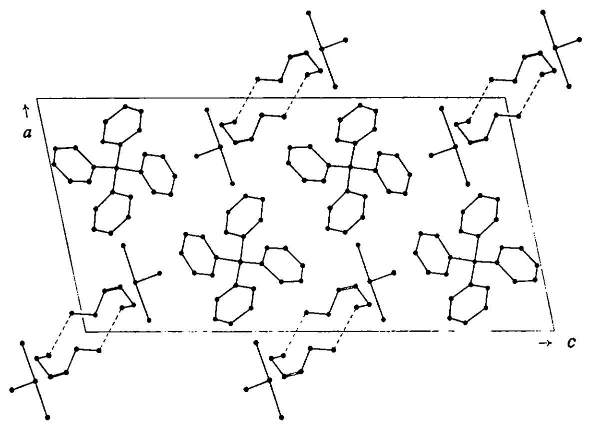

Fig. 5. Projection of the structure along the *b* axis. Dotted lines are intermolecular hydrogen bonds.

Table 5. P–C *bond lengths and* C–P–C *angles in the tetraphenylphosphonium cation*

Estimated standard deviations are given in parentheses

P——C (5)	1·82 (2) Å
P——C(11)	1·77 (2)
P——C(17)	1·82 (2)
P——C(23)	1·77 (2)
C(5)—P-C(11)	103·6 (11)°
C(5)—P-C(17)	113·6 (11)
C(5)—P-C(23)	111·4 (9)
C(11)-P-C(17)	109·5 (10)
C(11)-P-C(23)	112·6 (11)
C(17)-P-C(23)	106·2 (10)

Table 6. *Dihedral angles between planes in the tetraphenylphosphonium cation*

The planes $\pi(i)$ are defined by the phosphorus atom and two of the four carbon atoms bound to it: $\pi(1)$, [P C(5) C(11)]; $\pi(2)$, [P C(5) C(17)]; $\pi(3)$, [P C(5) C(23)]; $\pi(4)$, [P C(11) C(17)]; $\pi(5)$, [P C(11) C(23)]; $\pi(6)$, [P C(17) C(23)]. $\varphi(i)$ define the best planes through each phenyl group: $\varphi(1)$, [C(5)· · ·C(10)]; $\varphi(2)$, [C(11)· · ·C(16)]; $\varphi(3)$, [C(17)· · ·C(22)]; $\varphi(4)$, [C(23)· · ·C(28)].

$\pi(1) \wedge \pi(2)$		119°	$\varphi(1) \wedge \varphi(2)$		80°
$\pi(1)$	$\pi(3)$	121	$\varphi(1)$	$\varphi(3)$	58
$\pi(1)$	$\pi(4)$	122	$\varphi(1)$	$\varphi(4)$	62
$\pi(1)$	$\pi(5)$	120	$\varphi(2)$	$\varphi(3)$	72
$\pi(1)$	$\pi(6)$	88	$\varphi(2)$	$\varphi(4)$	71
$\pi(2)$	$\pi(3)$	120	$\varphi(3)$	$\varphi(4)$	80
$\pi(2)$	$\pi(4)$	115			
$\pi(2)$	$\pi(5)$	86			
$\pi(2)$	$\pi(6)$	123			
$\pi(3)$	$\pi(4)$	84			
$\pi(3)$	$\pi(5)$	116			
$\pi(3)$	$\pi(6)$	124			
$\pi(4)$	$\pi(5)$	118			
$\pi(4)$	$\pi(6)$	122			
$\pi(5)$	$\pi(6)$	120			

The P–C bond distances average 1·795 (10) Å, in good agreement with the sum of the covalent radii, and with the values reported in the literature (Goldstein, Seff & Trueblood, 1968, and references quoted therein).

The phenyl groups are not significantly non-planar and the 24 C–C aromatic bond distances average 1·41 (1) Å.

In Fig. 5 the structure is shown projected along the *b* axis.

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Die Kristallstrukturen von $K_4(PO_2NH)_44H_2O$ und $Cs_4\,(PO_2NH)_4.6H_2O$ und die Konformationsisomerie des ringförmigen Anions $(PO_2NH)_4^{4-}$

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The crystal structures of potassium tetrametaphosphimate tetrahydrate [$K_4(PO_2NH)_4 . 4H_2O$; $P2_1/c$; $a=6{\cdot}786$, $b=10{\cdot}371$, $c=14{\cdot}335$ Å; $\beta=57{\cdot}91°$; $Z=2$] and caesium tetrametaphosphimate hexahydrate [$Cs_4(PO_2NH)_4 . 6H_2O$; $P4_2/nmc$; $a=9{\cdot}693$, $c=11{\cdot}306$ Å; $Z=2$] were determined from three-dimensional X-ray diffraction intensities measured on an automatic diffractometer. The refinement yielded R values of 0·040 for the potassium salt and 0·055 for the caesium salt (1517 and 489 observed reflexions, respectively). The most interesting feature of the structures is the conformational isomerism of the anionic eight-membered P–N ring derived earlier from crystal vibrational spectra by Steger & Lunkwitz. In good agreement with their conclusions the ring in the potassium salt displays a chair form with almost $2/m$ symmetry and two opposite phosphorus atoms on the pseudo mirror plane. On the other hand the ring symmetry in the caesium salt is $\bar{4}2m$ (mirror planes through nitrogen atoms) and thus higher than $\bar{4}$ as derived from the spectra. The conformation is a saddle form.

Nach einer schwingungsspektroskopischen Analyse kristalliner Alkalisalze der Tetrametaphosphimsäure, $H_4(PO_2NH)_4$, nimmt der achtgliedrige P–N-Ring in unklarer Abhängigkeit vom Kation und der Hydratationszahl zwei verschiedene Konformationen an (Steger & Lunkwitz, 1969). Die eine ist eine Sesselform der Punktgruppe $2/m$ mit der Spiegelebene durch zwei im Ring gegenüberliegende Phosphoratome, die andere eine Sattelform der Punktgruppe $\bar{4}$, die vom Anion auch in wässriger Lösung bevorzugt wird.

Zur Prüfung dieser Aussagen und weiteren Untersuchung der Konformationsisomerie wurden die Kristallstrukturen von Kalium-tetrametaphosphimat-tetrahydrat, $K_4(PO_2NH)_4 . 4H_2O$, und Caesium-tetrametaphosphimat-hexahydrat, $Cs_4(PO_2NH)_4 . 6H_2O$, als bezeichnete Vertreter der Sessel- bzw. Sattelform bestimmt. Diese Strukturanalysen und ihre Ergebnisse werden nach zwei Kurzmitteilungen an anderen Stellen (Mootz & Berking, 1970; Berking & Mootz, 1970) im folgenden ausführlich beschrieben.

An Kristallstrukturen verwandter Substanzen wurden bisher die des Tetrametaphosphimsäure-dihydrats, $H_4(PO_2NH)_4 . 2H_2O$, (Migchelsen, Olthof & Vos, 1965: Punktgruppe des Rings exakt 2, angenähert $\bar{4}$, Wannenform) sowie des Natrium-trimetaphosphimat-tetrahydrats, $Na_3(PO_2NH)_3 . 4H_2O$, aufgeklärt (Olthof, Migchelsen & Vos, 1965: Ring in allgemeiner Lage, Sesselform).

Experimentelles und kristallographische Daten

Beide Salze bilden beim Eindunsten reiner wässriger Lösungen grosse farblose Kristalle (Lunkwitz & Steger, 1968). Die Raumgruppen wurden mit Weissenberg- und Präzessionsaufnahmen, die Gitterkonstanten diffraktometrisch und die Dichten durch Flotation bestimmt:

$K_4(PO_2NH)_4 . 4H_2O$	$Cs_4(PO_2NH)_4 . 6H_2O$
Raumgruppe: $P2_1/c$	Raumgruppe: $P4_2/nmc$
$a=6{,}786\ (2)$ Å;	$a=9{,}693\ (3)$ Å;
$b=10{,}371\ (3)$ Å;	
$c=14{,}335\ (4)$ Å;	$c=11{,}306\ (4)$ Å;
$\beta=57{,}91\ (2)°$;	
$V=854{,}7$ Å^3;	$V=1062{,}3$ Å^3;
$M=540{,}4$ g. Mol^{-1};	$M=951{,}7$ g. Mol^{-1};
$D_m=2{,}09$ g. cm^{-3};	$D_m=2{,}97$ g. cm^{-3};

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