Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent U values* ($\text{Å}^2 \times 10^3$) *with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
U	2500	2500	2500	33·8 (2)
Cl(1)	3463 (2)	4182 (2)	522 (2)	52 (1)
Cl(2)	2898 (2)	456 (2)	931 (3)	58 (1)
O(1)	3917 (4)	2173 (6)	3997 (6)	43 (3)
N(1)	4702 (5)	2395 (7)	6336 (8)	45 (4)
C(1)	4730 (5)	2319 (7)	4805 (9)	35 (4)
C(2)	5685 (6)	2408 (8)	3884 (10)	45 (4)
C(3)	5451 (7)	1712 (12)	2317 (12)	71 (6)
C(4)	6567 (7)	1686 (12)	4807 (13)	74 (6)
C(5)	5906 (8)	3951 (10)	3438 (13)	73 (6)
C(6)	3735 (7)	2204 (10)	7077 (10)	59 (5)
C(7)	5556 (8)	2772 (11)	7446 (11)	68 (6)

Table 2. *Bond lengths* (Å) *and angles* (°) *and their e.s.d.'s in parentheses*

Primed atoms are at $\tfrac{1}{2} - x, \tfrac{1}{2} - y, \tfrac{1}{2} - z$.

U–Cl(1)	2·609 (2)	N(1)–C(6)	1·48 (1)
U–Cl(2)	2·614 (2)	N(1)–C(7)	1·50 (1)
U–O(1)	2·246 (5)	C(1)–C(2)	1·54 (1)
O(1)–C(1)	1·268 (9)	C(2)–C(3)	1·56 (1)
C(1)–N(1)	1·30 (1)	C(2)–C(4)	1·55 (1)
		C(2)–C(5)	1·56 (1)
Cl(1)–U–Cl(2)	91·2 (1)	C(1)–N(1)–C(6)	118·8 (6)
Cl(1)–U–Cl(2)'	88·8 (1)	C(1)–N(1)–C(7)	126·4 (7)
O(1)–U–Cl(1)	89·4 (1)	C(6)–N(1)–C(7)	114·6 (7)
O(1)–U–Cl(2)	89·2 (2)	C(1)–C(2)–C(4)	113·6 (7)
U–O(1)–C(1)	165·2 (5)	C(1)–C(2)–C(3)	107·6 (7)
O(1)–C(1)–N(1)	118·5 (7)	C(1)–C(2)–C(5)	105·9 (7)
O(1)–C(1)–C(2)	116·7 (7)	C(3)–C(2)–C(4)	107·9 (8)
N(1)–C(1)–C(2)	124·8 (6)	C(3)–C(2)–C(5)	108·6 (7)
		C(4)–C(2)–C(5)	113·1 (7)

Preez, Gellatly, Jackson, Nassimbeni & Rodgers, 1978) (hmpa = hexamethylphosphoramide), and differ from UCl₄(tppo)₂ (Bombieri, Brown & Graziani, 1975) (tppo = triphenylphosphine oxide) in which the neutral ligands are *cis* related. The U—Cl distances of 2·609 (2) and 2·614 (2) Å, equal within the limits of the errors, are usual values for these complexes regardless of their *cis* or *trans* geometry. The U—O distance, 2·246 (5) Å, is in agreement with the values observed in UCl₄-
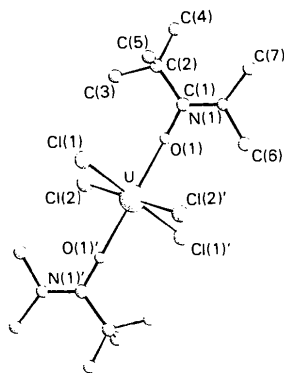

Fig. 1. View of the molecule down **b**. The primed atoms are centrosymmetrically related to those without primes through the U atom.

(hmpa)₂ [2·23 (1) Å], UCl₄ (tppa)₂ [2·226 (6) Å] and in UCl₄ (tppo)₂ [2·242 (7) Å].

The dimensions of the neutral ligands are not unusual. The crystal packing is mainly determined by van der Waals forces and all intermolecular contacts agree with those predicted from the sums of the ionic radii.

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trans-Hydrido(phenyl)bis(triisopropylphosphine)platinum(II), [Pt(C₆H₅)(C₉H₂₁P)₂H]

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Abstract. $M_r = 593·7$, triclinic, $P\bar{1}$, $a = 15·997$ (6), $b = 19·803$ (5), $c = 10·109$ (3) Å, $\alpha = 116·48$ (1), $\beta = 106·28$ (1), $\gamma = 82·62$ (1)°, $U = 2751·5$ Å³, $Z = 4$, $D_x = 1·43$ Mg m⁻³, $F(000) = 1200$, Mo $K\bar{\alpha}$, $\mu = 5·00$ mm⁻¹, $T = 293$ (2) K, $R = 0·078$ for 7153 unique reflections from four rapidly degrading crystals. Independent molecules are dimensionally similar but

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differ in the relative orientations of the phosphine ligands about the P···P axis. Principal bond lengths and angles (average values) are Pt–P 2·272 (5), Pt–C 2·07 (2) Å, P–Pt–P 168·8 (2) and P–Pt–C 94·6 (3) and 96·3 (3)°.

Introduction. The fact that transition-metal–phosphine bond lengths are subject to steric as well as electronic control is well established (Tolman, 1977; Mason & Meek, 1978; Mazid, Russell & Tucker, 1980). For trans-planar $Pt^{II}L_2X_2$ and $Pt^{II}L_2XY$ complexes (L = tertiary phosphine) the important steric interactions are those between the anionic ligands and the phosphine-ligand substituents. Hence, systematic variation of the X ligands will cause systematic variation of the $M–L$ distances. For large-cone-angle phosphines (Tolman, 1977) the $M–L$ bond compressions can be substantial, e.g. Pt–P is 2·25 (1) Å in $Pt(Pcy_3)_2H_2$ (Immirzi, Musco, Carturan & Belluco, 1975) and 2·371 (2) Å in $Pt(Pcy_3)_2I_2$ (Alcock & Leviston, 1974) (cy = cyclohexyl). Because little comparable information is available for complexes with small- and medium-sized phosphines we have begun a systematic study of the $trans$-$Pt^{II}(P^iPr_3)_2$-X_2/XY system. We report, here, on the hydridophenyl derivative and compare its coordination geometry with those of the dichloro and hydridochloro derivatives.

Experimental. Colourless parallelepipedal crystals from ethanol/water, triclinic, $P\bar{1}$ (confirmed by structure solution). The reduced cell has dimensions $a = 10·109$, $b = 15·997$, $c = 17·772$ Å, $\alpha = 89·06$, $\beta = 85·87$, $\gamma = 73·72°$ and is related to the cell used in this work by the transformation $a_{reduced} = Ta$ where $t_{11} = 0$, $t_{12} = 0$, $t_{13} = 1$, $t_{21} = -1$, $t_{22} = 0$, $t_{23} = 0$, $t_{31} = 0$, $t_{32} = 1$ and $t_{33} = 1$. Bounding forms {001}, {010} and {100}; Picker FACS-I diffractometer, Mo K radiation, $2\theta_{max}$ = 55°; data aggregated from four crystals,* average degradation rate ~20%/1000 measured reflections, maximum allowed degradation ~80%; 21 521 reflections including standards (3 every 97 data, indices $00\bar{5}$, 900 and 0,10,0); 7153 unique data [$I \geq 3\sigma(I)$], corrected for absorption (de Meulenaer & Tompa, 1965) and crystal degradation (Churchill & Kalra, 1974);* data from different crystals scaled according to intensities of standards, $R_{int}(=\sum|F_o^2 - \langle F_o^2\rangle|/\sum F_o^2)$ = 0·096, $R_s[=\sum\sigma_s(F_o)/\sum|F_o|]$ = 0·033 (Robertson & Whimp, 1975); cell dimensions (four-crystals average) from 12 reflections each with $2\theta > 39°$ [$\lambda(Mo K\alpha_1)$

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* Lists of structure factors and anisotropic thermal parameters and a table specifying data-collection and -reduction details (including crystal dimensions, degradation rates, transmission factors etc.) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38702 (44 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

= 0·70926 Å]; structure solved from Patterson and Fourier syntheses; refined by full-matrix least squares minimizing $\sum w(|F_o| - |F_c|)^2$ with $w = [\sigma_s^2(F_o) + 0·002|F_o|^2]^{-1}$; anisotropic thermal parameters for Pt, P and phenyl C atoms only; H atoms not discernible in difference maps and not included in scattering model; $R = 0·078$; $R_w = 0·109$; $(\Delta/\sigma)_{max} = 0·2$; $(\Delta\rho)_{max} = 2·2\,e\,Å^{-3}$ near Pt and $1·3\,e\,Å^{-3}$ elsewhere; atomic scattering amplitudes, with dispersion corrections for Pt, from *International Tables for X-ray Crystallography* (1974); no correction for secondary extinction; computer programs: ANUCRYS (McLaughlin, Taylor & Whimp, 1977), SHELX76 (Sheldrick, 1976), PLUTO78 (Motherwell, 1978).
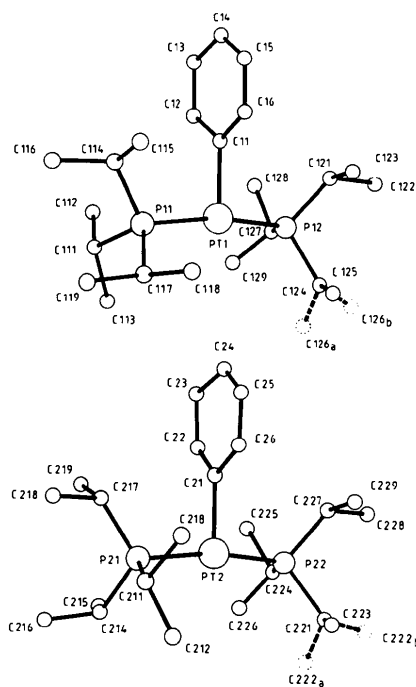

Fig. 1. Conformation and atom numbering in the two crystallographically independent molecules of the title compound. Dashed atoms indicate 50% site occupancy.
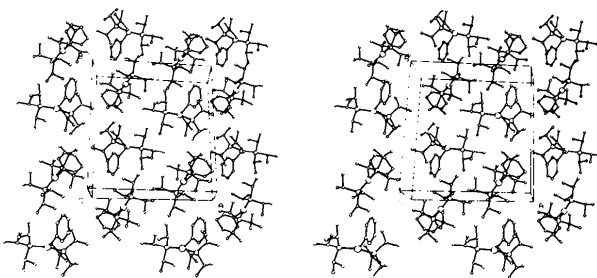

Fig. 2. The crystal packing arrangement viewed approximately parallel to c. Both disordered C sites are shown.

Discussion. Crystals of trans-Pt(P^{i}Pr$_3$)$_2$HPh (1) are composed of discrete molecules, two per asymmetric unit, separated by normal van der Waals contacts. The molecular geometry and atom numbering are shown in Fig. 1 and the crystal packing arrangement, viewed approximately along c, is shown by the stereopair of Fig. 2. Atomic coordinates are listed in Table 1 and bond lengths and interbond angles are listed in Table 2. The tabulated e.s.d.'s are likely to be underestimates since errors in the form of the crystal-degradation correction will result in systematic errors in the data.

The Pt—P bond lengths in (1) do not differ significantly. The mean value [2·272 (5) Å] is significantly less (shorter) than that in trans-Pt(P^{i}Pr$_3$)$_2$Cl$_2$

Table 1. *Final atomic and thermal parameters (with estimated standard deviations in parentheses)*

	x	y	z	U_{eq} or U(Å^2)
Pt(1)	0·27831 (4)	0·39545 (4)	0·20129 (7)	0·04207†
Pt(2)	0·25721 (4)	−0·08200 (4)	0·19784 (7)	0·04352†
P(11)	0·4009 (3)	0·3911 (2)	0·1263 (5)	0·0417†
P(12)	0·1694 (3)	0·4190 (3)	0·3200 (5)	0·0463†
P(21)	0·3200 (3)	0·0052 (2)	0·1669 (5)	0·0466†
P(22)	0·2107 (3)	−0·1584 (3)	0·2754 (5)	0·0505†
C(11)	0·204 (1)	0·330 (2)	−0·020 (2)	0·068†
C(12)	0·160 (1)	0·367 (2)	−0·117 (3)	0·097†
C(13)	0·110 (2)	0·319 (3)	−0·272 (4)	0·132†
C(14)	0·114 (2)	0·234 (3)	−0·312 (4)	0·126†
C(15)	0·156 (2)	0·202 (2)	−0·223 (3)	0·110†
C(16)	0·201 (2)	0·249 (2)	−0·075 (3)	0·088†
C(21)	0·180 (2)	−0·126 (1)	−0·021 (2)	0·073†
C(22)	0·095 (2)	−0·105 (2)	−0·063 (3)	0·113†
C(23)	0·038 (2)	−0·133 (4)	−0·224 (8)	0·254†
C(24)	0·089 (4)	−0·198 (4)	−0·325 (8)	0·222†
C(25)	0·168 (3)	−0·221 (2)	−0·297 (3)	0·140†
C(26)	0·217 (2)	−0·190 (1)	−0·148 (2)	0·086†
C(111)	0·427 (1)	0·484 (1)	0·144 (2)	0·069 (5)
C(112)	0·348 (2)	0·511 (1)	0·051 (3)	0·091 (6)
C(113)	0·445 (2)	0·543 (2)	0·316 (3)	0·113 (8)
C(114)	0·396 (1)	0·328 (1)	−0·081 (2)	0·063 (4)
C(115)	0·405 (2)	0·246 (1)	−0·110 (3)	0·094 (7)
C(116)	0·459 (2)	0·348 (1)	−0·152 (3)	0·096 (7)
C(117)	0·499 (2)	0·365 (1)	0·248 (3)	0·101 (7)
C(118)	0·486 (2)	0·300 (1)	0·278 (3)	0·092 (6)
C(119)	0·587 (2)	0·361 (2)	0·198 (3)	0·110 (8)
C(121)	0·083 (1)	0·349 (1)	0·226 (2)	0·078 (5)
C(122)	−0·001 (2)	0·366 (2)	0·305 (3)	.0·115 (8)
C(123)	0·119 (2)	0·273 (2)	0·218 (4)	0·131 (10)
C(124)	0·207 (2)	0·432 (2)	0·518 (3)	0·116 (8)
C(125)	0·271 (2)	0·359 (2)	0·524 (3)	0·118 (9)
C(126a)*	0·226 (3)	0·516 (3)	0·637 (6)	0·103 (15)
C(126b)*	0·163 (4)	0·469 (4)	0·634 (8)	0·138 (21)
C(127)	0·112 (1)	0·507 (1)	0·336 (2)	0·080 (6)
C(128)	0·060 (2)	0·499 (2)	0·181 (4)	0·155 (12)
C(129)	0·172 (2)	0·570 (2)	0·389 (3)	0·121 (9)
C(211)	0·434 (1)	−0·024 (1)	0·147 (2)	0·077 (5)
C(212)	0·494 (2)	−0·033 (1)	0·284 (3)	0·096 (7)
C(213)	0·428 (2)	−0·102 (1)	−0·001 (3)	0·102 (7)
C(214)	0·338 (2)	0·097 (1)	0·347 (3)	0·100 (7)
C(215)	0·256 (2)	0·123 (2)	0·400 (3)	0·118 (9)
C(216)	0·377 (2)	0·160 (2)	0·336 (3)	0·121 (9)
C(217)	0·266 (1)	0·019 (1)	−0·005 (2)	0·070 (5)
C(218)	0·324 (1)	0·059 (1)	−0·057 (3)	0·086 (6)
C(219)	0·180 (2)	0·062 (1)	0·009 (3)	0·096 (7)
C(221)	0·299 (2)	−0·188 (2)	0·416 (3)	0·109 (8)
C(222a)*	0·302 (4)	−0·129 (3)	0·580 (6)	0·115 (17)
C(222b)*	0·287 (4)	−0·233 (3)	0·473 (7)	0·118 (17)
C(223)	0·384 (2)	−0·213 (2)	0·355 (3)	0·122 (9)
C(224)	0·129 (2)	−0·116 (1)	0·381 (3)	0·094 (7)
C(225)	0·049 (2)	−0·096 (2)	0·275 (4)	0·154 (12)
C(226)	0·155 (2)	−0·042 (2)	0·504 (5)	0·162 (13)
C(227)	0·160 (2)	−0·248 (1)	0·122 (3)	0·091 (6)
C(228)	0·106 (2)	−0·292 (2)	0·178 (4)	0·140 (11)
C(229)	0·226 (2)	−0·302 (2)	0·043 (4)	0·143 (11)

* Atoms with site occupancy of 0·5.

† $U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{23}\cos\alpha + 2U_{13}\cos\beta + 2U_{12}\cos\gamma)$.

Table 2. *Bond lengths (Å) and angles (°) with estimated standard deviations in parentheses*

	n = 1	n = 2
Pt(n)·P(n1)	2·272 (4)	2·279 (4)
Pt(n)—P(n2)	2·271 (4)	2·265 (5)
Pt(n)·C(n1)	2·10 (2)	2·05 (2)
P(n1)—C(n11)	1·86 (2)	1·88 (2)
P(n1)·C(n14)	1·88 (2)	1·89 (3)
P(n1)·C(n17)	1·88 (2)	1·83 (2)
P(n2)—C(n21)	1·83 (2)	1·94 (3)
P(n2)—C(n24)	1·83 (2)	1·80 (2)
P(n2)—C(n27)	1·82 (2)	1·86 (2)
		Mean 1·86 (4)
C(n1)—C(n2)	1·45 (3)	1·38 (4)
C(n2)—C(n3)	1·48 (4)	1·51 (6)
C(n3)—C(n4)	1·54 (6)	1·54 (10)
C(n4)—C(n5)	1·30 (5)	1·29 (6)
C(n5)—C(n6)	1·39 (3)	1·37 (4)
C(n6)—C(n1)	1·45 (3)	1·54 (3)
		Mean 1·44 (9)
C(n11)—C(n12)	1·54 (3)	1·52 (3)
C(n11)—C(n13)	1·57 (3)	1·59 (3)
C(n14)—C(n15)	1·52 (3)	1·51 (3)
C(n14)—C(n16)	1·57 (3)	1·54 (3)
C(n17)—C(n18)	1·50 (3)	1·61 (3)
C(n17)—C(n19)	1·59 (3)	1·53 (3)
C(n21)—C(n22)	1·67 (3)	1·54 (6), 1·31 (6)*
C(n21)—C(n23)	1·51 (4)	1·57 (4)
C(n24)—C(n25)	1·69 (4)	1·56 (4)
C(n24)—C(n26)	1·40 (7), 1·56 (6)*	1·45 (4)
C(n27)—C(n28)	1·50 (4)	1·63 (4)
C(n27)—C(n29)	1·49 (3)	1·52 (4)
		Mean 1·55 (6)
P(n1)—Pt(n)—P(n2)	168·6 (2)	169·0 (2)
P(n1)—Pt(n)—C(n1)	94·9 (4)	94·4 (5)
P(n2)—Pt(n)—C(n1)	96·1 (4)	96·6 (5)
Pt(n)—P(n1)—C(n11)	112·2 (6)	110·8 (7)
Pt(n)·P(n1)—C(n14)	115·6 (6)	110·6 (8)
Pt(n)—P(n1)—C(n17)	111·8 (8)	115·4 (6)
Pt(n)—P(n2)—C(n21)	115·6 (7)	115·3 (8)
Pt(n)—P(n2)—C(n24)	113·9 (9)	113·8 (8)
Pt(n)—P(n2)—C(n27)	112·7 (7)	116·2 (8)
C(n11)—P(n1)—C(n14)	101·9 (8)	102·3 (10)
C(n11)—P(n1)—C(n17)	105·1 (10)	103·8 (9)
C(n14)—P(n1)—C(n17)	109·3 (10)	112·9 (10)
C(n21)—P(n2)—C(n24)	106·6 (11)	102·0 (12)
C(n21)—P(n2)—C(n27)	103·8 (9)	104·4 (11)
C(n24)—P(n2)—C(n27)	103·0 (12)	103·4 (11)
Pt(n)—C(n1)—C(n2)	119 (2)	124 (2)
Pt(n)—C(n1)—C(n6)	120 (2)	119 (2)
C(n6)—C(n1)—C(n2)	122 (2)	117 (2)
C(n1)—C(n2)—C(n3)	117 (3)	125 (4)
C(n2)—C(n3)—C(n4)	114 (3)	106 (3)
C(n3)—C(n4)—C(n5)	127 (4)	132 (5)
C(n4)—C(n5)—C(n6)	118 (4)	117 (5)
C(n5)—C(n6)—C(n1)	122 (3)	121 (3)
		Mean 120 (6)
P(n1)—C(n11)—C(n12)	109·1 (14)	113·6 (15)
P(n1)—C(n11)—C(n13)	109·9 (15)	107·6 (14)
P(n1)—C(n14)—C(n15)	110·6 (14)	110·9 (17)
P(n1)—C(n14)—C(n16)	116·4 (13)	114·7 (18)
P(n1)—C(n17)—C(n18)	114·7 (17)	115·9 (13)
P(n1)—C(n17)—C(n19)	113·5 (17)	111·6 (15)
P(n2)—C(n21)—C(n22)	117·0 (15)	108 (3), 126 (3)*
P(n2)—C(n21)—C(n23)	110·6 (17)	109·1 (19)
P(n2)—C(n24)—C(n25)	108·7 (19)	107·8 (20)
P(n2)—C(n24)—C(n26)	127 (3), 114 (3)*	112·6 (21)
P(n2)·C(n27)·C(n28)	109·6 (18)	114·3 (18)
P(n2)—C(n27)—C(n29)	112·7 (16)	111·6 (18)
		Mean 111·9 (28)
C(n12)—C(n11)—C(n13)	109 (2)	109 (2)
C(n15)—C(n14)—C(n16)	111 (2)	111 (2)
C(n18)—C(n17)—C(n19)	114 (2)	109 (2)
C(n22)—C(n21)—C(n23)	106 (2)	122 (3), 110 (3)*
C(n25)—C(n24)—C(n26)	117 (3), 124 (3)*	101 (3)
C(n28)—C(n27)—C(n29)	104 (2)	110 (2)
		Mean 108 (4)

* These quantities refer to the disordered atoms and the mean values given below do not include them.

(2) [2·340 (2) Å] but agrees well with that in *trans*-Pt(P^{i}Pr$_3$)$_2$HCl (3) [2·287 (1) Å] (Robertson & Tucker, 1982). P—Pt—P angles in (1) and (3) also agree well [168·8 (2)° av. in (1), and 171·16 (3)° in (3)]. In contrast, the corresponding angle in (2) is symmetry constrained to 180°. Differences between (2) and (3) are attributable, in detail, to steric effects (Robertson & Tucker, 1982, 1983). The close similarity between the Pt—P distances and P—Pt—P angles in (1) and (3) reflects, primarily, the fact that the van der Waals thickness of an aromatic ring is approximately the same as the van der Waals diameter of a Cl atom. The Pt—C distance [av. 2·07 (2) Å] agrees well with that in the (σ-cyclohexyl)hydridobis(triphenylphosphine) analogue *trans*-Pt(PPh$_3$)$_2$H(σC$_6$H$_9$) [2·08 (1) Å] (Bilton & Robertson, 1982) but, reflecting both the differing steric requirements and differing σ-orbital radii of sp^2 cf. sp^3 hybridized donor C atoms, is appreciably shorter than that in *trans*-Pt(PPh$_3$)$_2$H(CH$_2$CN) [2·15 (1) Å] (Ros, Michelin, Belluco, Zanotti, Del Pra & Bombieri, 1978). Other bond lengths and bond angles are insufficiently well determined to warrant discussion.

The ligating phenyl group in each molecule is approximately perpendicular to the Pt-coordination plane, as is required to avoid overcrowding in that plane. The conformational similarity of the right-hand sides of molecules 1 and 2 in Fig. 1 is self evident (even to the extent of the isopropyl-group disorder). The molecules differ principally in the orientation of the left-hand phosphine group. In molecule 1 the P—C bonds of the two phosphines are approximately eclipsed when viewed along the P—P vector whereas in molecule 2 they are approximately staggered. The present result supports our earlier contention (Bennett, Ho, Jeffery, McLaughlin & Robertson, 1982) that energy differences between eclipsed and staggered forms are small.

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Structure of the Pentahydrate of the Samarium(III) Perchlorate Complex with the Crown Polyether 1,4,7,10,13-Pentaoxacyclopentadecane, [Sm(C$_{10}$H$_{20}$O$_5$)$_2$(H$_2$O)$_4$]$^{3+}$. 3ClO$_4^-$.H$_2$O

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Abstract. $M_r = 979.30$, monoclinic, $P2_1/c$, $a = 16.150$ (5), $b = 14.898$ (5), $c = 21.055$ (5) Å, $\beta = 129.37$ (3)°, $V = 3916.27$ Å^3, $Z = 4$, $D_m = 1.62$ (3), $D_x = 1.66$ Mg m^{-3} (by flotation), λ(Mo $K\alpha$) = 0.71069 Å, μ(Mo $K\alpha$) = 1.83 mm^{-1}, $F(000) = 1996$, $T = 296$ (4) K, $R(F) = 0.067$ for 3540 observed reflections. The samarium(III) ion is found to be coordinated to nine oxygen atoms, five from one of the ether molecules and the remaining four from water molecules. The second ether molecule does not take part in coordination. Three of the water molecules link to the second ether ring by forming hydrogen bonds with the