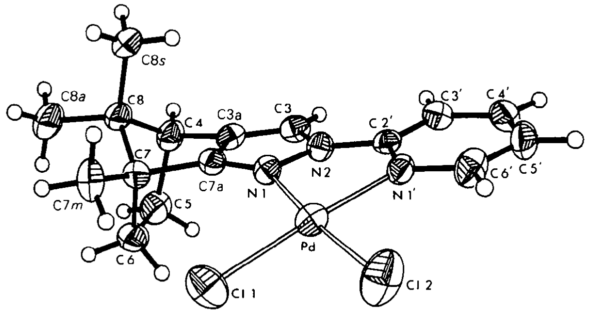

Fig. 1. Perspective view and atom labelling of the title complex. Thermal ellipsoids are drawn at the 35% probability level; H atoms with 0·15 Å radii.

The study confirms the structure of the ligand and its absolute configuration (as determined by anomalous dispersion) is consistent with that of (+)-camphor (Northolt & Palm, 1966) from which it was synthesized. The pyrazole and pyridine rings are both planar (mean deviations 0·007 and 0·010 Å respectively) and only slightly inclined [5·3 (4)°] to one another.

Coordination about Pd is essentially planar with no significant pyramidal distortion as is observed in Pd^{II} complexes with sterically encumbered ligands (Newkome, Fronczek, Gupta, Puckett, Pantaleo & Kiefer, 1982). Indeed the N(1), N(1'), Pd and Cl(2) atoms are coplanar (mean deviation 0·004 Å) with Cl(1) slightly displaced [0·188 (2) Å] from the plane, probably due to a weak C(7*m*)methyl···Cl(1) interaction. Also, the Pd—N bonds [2·039 (5) and 2·026 (5) Å] show no significant lengthening due to steric interactions as is the case with more bulky ligands (Deeming, Rothwell, Hursthouse & Backer-Dirks, 1979; Newkome *et al.*, 1982). Indeed the parameters defined by Newkome *et al.* (1982) to describe molecular distortions in such square-planar complexes [out-of-plane distortions: $d_1 = 0{\cdot}203$ (2), $d_2 = 0{\cdot}025$ (2) Å; ring tilts: $\beta_1 = 9{\cdot}3$ (2), $\beta_2 = 6{\cdot}0$ (2); ring coplanarity distortions: $\gamma_1 = 5{\cdot}8$ (5), $\gamma_2 = 1{\cdot}7$ (5)°] are all considerably less than those observed in dichloro(6-methyl-2,2'-bipyridine)palladium(II).

In conclusion, the chiral ligand (II) appears to offer little steric resistance to coordination and hence it and related ligands currently under study should prove useful chiral auxiliaries in synthesis.

References

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.
Deeming, A. J., Rothwell, I. P., Hursthouse, M. B. & Backer-Dirks, J. D. J. (1979). *J. Chem. Soc. Chem. Commun.* pp. 670–672.
Jacquier, R. & Maury, G. (1967). *Bull. Soc. Chim. Fr.* pp. 295–297.
Khan, M. A. & Polya, J. B. (1970). *J. Chem. Soc. C*, pp. 85–91.
Newkome, G. R., Fronczek, F. R., Gupta, V. K., Puckett, W. E., Pantaleo, D. C. & Kiefer, G. E. (1982). *J. Am. Chem. Soc.* **104**, 1782–1783.
Northolt, M. G. & Palm, J. H. (1966). *Recl Trav. Chim. Pays-Bas*, **85**, 143–146.
Sheldrick, G. M. (1981). *SHELXTL User Manual*, revision 3. Nicolet XRD Corporation, Cupertino, California.

Acta Cryst. (1983). C**39**, 1625–1628

5,10,15,20-Tetraphenylporphyrin–Bis(trifluoroacetic acid)–Dioxouranium(VI) Trifluoroacetate, $C_{44}H_{30}N_4.2C_2HF_3O_2.[UO_2][C_2F_3O_2]_2$

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Abstract. $M_r = 1339$, monoclinic, Pc, $a = 9{\cdot}593$ (8), $b = 17{\cdot}481$ (4), $c = 15{\cdot}816$ (9) Å, $\beta = 97{\cdot}3$ (2)°, $Z = 2$, $V = 2631$ (7) Å^3, $D_x = 1{\cdot}69$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 9{\cdot}85$ mm^{-1}, $T = 233$ (5) K, $F(000) = 1304$, final $R = 0{\cdot}068$ for 3417 observed reflections. The structural features generate a pseudosymmetry which renders the interpretation of the Fourier synthesis and refinement of the structure difficult. The uranyl group has no direct contact with the tetraphenylporphyrin (TPP) but presents a pentacoordination in the equatorial plane with five O atoms belonging to four trifluoroacetate groups. Hydrogen bonds connect the TPP and the bridging trifluoroacetic acid molecules (Htfa).

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Introduction. The present work is part of a research programme which is being carried out on uranium–macrocycle complexes. Single crystals were supplied by Drs G. Folcher and G. de Villardi (Département de Physicochimie, CEN–Saclay). The space group was determined photographically. After an unsuccessful trial of structure refinement with data collected at room temperature, the intensities were measured at 233 ±5 K. Cell parameters at room temperature: $a = 9{\cdot}601\,(2)$, $b = 17{\cdot}473\,(3)$, $c = 15{\cdot}988\,(4)$ Å, $\beta = 98{\cdot}2\,(2)°$, $V = 2655\,(2)$ Å^3.

Experimental. A blue prismatic crystal (approximate dimensions 0·075 × 0·075 × 0·30 mm) with boundary faces {011} was selected and mounted along the [100] direction. Enraf–Nonius CAD-4 diffractometer equipped with a cooling device. Unit-cell dimensions and their estimated standard deviations obtained from a least-squares fit of the optimized diffractometer setting angles of 25 selected reflections. 3792 unique reflections ($2\theta < 116°$) measured with the $\omega/2\theta$ scan technique [scan angle $(1{\cdot}5 + 0{\cdot}14\,\mathrm{tg}\theta)°$], graphite-monochromatized Cu$K\alpha$ radiation; 366 considered unobserved [$I < 1{\cdot}5\sigma(I)$]. Three standard reflections monitored every 100 measurements: no significant fluctuations. Data corrected for Lorentz–polarization effects; absorption corrections made taking crystal morphology into account. The position of the U atom was determined from a Patterson synthesis. The particular position of this atom and of the centre of the tetraphenylporphyrin added a non-existing twofold axis to the Fourier map, whose symmetry group was then $P2/c$. The problem of separating the real structure from its pseudosymmetry-generated image was difficult, since pseudosymmetric atomic positions were very close to those of the true atoms and the tendency to produce centrosymmetric phases was very strong. The noncentrosymmetric phases computed from the positions of the U atom and the C atoms of one phenyl group located in a Fourier map were used as the initial step to break the pseudosymmetry. The subsequent Fourier synthesis calculated with the 1790 highest $|F_o|$ using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) enabled us to locate the TPP and three tfa groups. At this stage the R value was 0·28. Difference maps, based on all observed reflections, calculated with the *SHELX* program (Sheldrick, 1976), revealed the locations of the missing tfa group and a number of small peaks in the neighbourhood of the F atoms which were attributed to secondary positions of these atoms. The H atoms were placed respecting stereochemistry. The structure was refined by full-matrix least squares to $R = 0{\cdot}068$ using *SHELX*; $\sum w(\Delta F)^2$ minimized, unit weights. Only thermal parameters of U were refined anisotropically. Population and isotropic thermal parameters of the F atoms were estimated from a difference map; only the thermal parameters of those F atoms with occupancy greater than 0·5 were refined. Bond-length constraints with a sufficiently large e.s.d. (0·02) were applied to the C—O, C—C and C—N distances. The trifluoromethyl, carboxyl and phenyl groups were refined as rigid groups. The ratio of observed reflexions to refined parameters was 12. Final $\Delta\rho$ excursions $\leq 0{\cdot}6$ e Å^{-3} except for a small region around the U atom and one of the CF_3 groups; $(\Delta/\sigma)_{\max} = 0{\cdot}6$; no correction for secondary extinction. Values for the atomic scattering factors and anomalous-dispersion terms for U^{VI} from *International Tables for X-ray Crystallography* (1974) and neutral-atom scattering factors for C, N, O, F and H were those incorporated in the programs.

Discussion. The final positional parameters, thermal parameters and occupancy factors are in Table 1.*

The crystal structure (Fig. 1) is built from UO_2-(tfa)$_2$(Htfa)$_2$ units and TPP molecules which alternate along the [100] direction. The short distances from O(1) to N(1) [2·86 (2)] and N(3) [2·90 (2) Å], O(4′) to N(2) [2·91 (2)] and N(4) [2·88 (2) Å], and angles at central H atoms [153 (1), 157 (1), 145 (1), 159 (1)° respectively] indicate hydrogen bonds connecting adjacent groups.

The uranyl cation is quasilinear, the O(9)—U—O(10) atoms forming an angle of 178·4 (0·9)°; the U—O(9) and U—O(10) distances are respectively 1·70 (2) and 1·81 (2) Å. The unusual asymmetry in these distances could be related to the different contact distances between the two uranyl O and porphyrin N atoms [N(1)—O(9) 2·82 (2), N(2′)—O(10) 2·94 (2) Å].

The uranyl is coordinated to five O atoms: the O(2) and O(3) of two monodentate Htfa bridging the uranyl

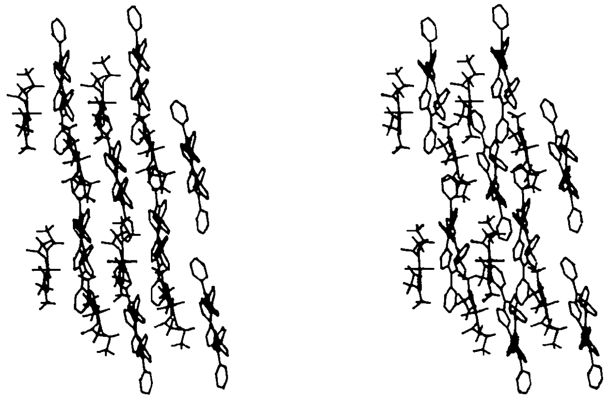

Fig. 1. The crystal packing (*ORTEP*II, Johnson, 1971).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and angles involving the TPP atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38758 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates, equivalent isotropic or isotropic temperature factors* (Å^2 × 10^3), *and occupancy factors* (k)

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

	x	y	z	k	$U_{\rm eq}/U$
U	1·000	0·16409 (5)	$\frac{1}{4}$	1·0	60 (1)
O(9)	0·8814 (19)	0·1507 (11)	0·3200 (11)	1·0	63 (6)
O(10)	1·1297 (23)	0·1794 (13)	0·1778 (14)	1·0	87 (8)
O(1)	0·6956 (14)	0·3114 (7)	0·2088 (13)	1·0	63 (5)
O(2)	0·9277 (14)	0·2893 (7)	0·2097 (13)	1·0	85 (6)
C(1)	0·8141 (14)	0·3284 (7)	0·1996 (13)	1·0	52 (5)
C(2)	0·8472 (14)	0·4091 (6)	0·1657 (8)	1·0	79 (8)
F(21)	0·9046 (14)	0·4085 (6)	0·0918 (8)	0·7	71 (10)
F(22)	0·7268 (14)	0·4502 (6)	0·1479 (8)	0·7	77 (10)
F(23)	0·9260 (14)	0·4470 (6)	0·2259 (8)	0·7	92 (10)
F(24)	0·7941 (14)	0·4607 (6)	0·1904 (8)	0·15	100
F(25)	0·9847 (14)	0·4213 (6)	0·1822 (8)	0·15	100
F(26)	0·8097 (14)	0·4077 (6)	0·0818 (8)	0·15	100
F(27)	0·9698 (14)	0·4087 (6)	0·1294 (8)	0·15	90
F(28)	0·7411 (14)	0·4298 (6)	0·1076 (8)	0·15	90
F(29)	0·8583 (14)	0·4591 (6)	0·2299 (8)	0·15	90
O(3)	1·1347 (17)	0·2460 (12)	0·3538 (8)	1·0	80 (5)
O(4)	1·3402 (17)	0·2937 (12)	0·3279 (8)	1·0	58 (4)
C(3)	1·2482 (17)	0·2810 (12)	0·3706 (8)	1·0	55 (6)
C(4)	1·2713 (21)	0·3152 (10)	0·4619 (7)	1·0	85 (9)
F(41)	1·1595 (21)	0·3192 (10)	0·5037 (7)	0·4	90
F(42)	1·3206 (21)	0·3860 (10)	0·4594 (7)	0·4	90
F(43)	1·3659 (21)	0·2724 (10)	0·5091 (7)	0·4	90
F(44)	1·4054 (21)	0·3122 (10)	0·4960 (7)	0·3	100
F(45)	1·1978 (21)	0·2766 (10)	0·5141 (7)	0·3	100
F(46)	1·2329 (21)	0·3882 (10)	0·4638 (7)	0·3	100
F(47)	1·2814 (21)	0·2588 (10)	0·5181 (7)	0·3	100
F(48)	1·1656 (21)	0·3604 (10)	0·4766 (7)	0·3	100
F(49)	1·3881 (21)	0·3570 (10)	0·4779 (7)	0·3	100
O(5)	0·8529 (34)	0·1249 (10)	0·1438 (11)	1·0	91 (6)
O(6)	0·8440 (34)	0·2035 (10)	0·0285 (11)	1·0	151 (10)
C(5)	0·8341 (34)	0·1412 (10)	0·0659 (11)	1·0	93 (6)
C(6)	0·8085 (23)	0·0729 (9)	0·0089 (11)	1·0	110 (11)
F(61)	0·6835 (23)	0·0423 (9)	0·0153 (11)	0·7	125 (11)
F(62)	0·9003 (23)	0·0154 (9)	0·0195 (11)	0·7	125 (11)
F(63)	0·8046 (23)	0·0899 (9)	−0·0740 (11)	0·7	139 (11)
F(64)	0·9163 (23)	0·0644 (9)	−0·0344 (11)	0·3	150
F(65)	0·7872 (23)	0·0064 (9)	0·0461 (11)	0·3	150
F(66)	0·6954 (23)	0·0824 (9)	−0·0477 (11)	0·3	150
O(7)	1·0526 (31)	0·0212 (9)	0·2520 (15)	1·0	102 (7)
O(8)	1·1861 (31)	0·0849 (9)	0·3490 (15)	1·0	117 (8)
C(7)	1·1361 (31)	0·0293 (9)	0·3138 (15)	1·0	95 (10)
C(8)	1·1929 (23)	−0·0469 (9)	0·3545 (13)	1·0	158 (17)
F(81)	1·0981 (23)	−0·0932 (9)	0·3826 (13)	0·5	81 (11)
F(82)	1·3318 (23)	−0·0536 (9)	0·3588 (13)	0·5	104 (11)
F(83)	1·2618 (23)	−0·0914 (9)	0·3053 (13)	0·5	144 (12)
F(84)	1·1664 (23)	−0·0522 (9)	0·4348 (13)	0·5	157 (12)
F(85)	1·2842 (23)	−0·0317 (9)	0·4229 (13)	0·5	152 (12)
F(86)	1·1386 (23)	−0·1085 (9)	0·3143 (13)	0·5	159 (13)
N(1)	0·6009 (19)	0·1999 (10)	0·3213 (10)	1·0	45 (4)
C(9)	0·6471 (24)	0·1951 (11)	0·4070 (11)	1·0	56 (6)
C(10)	0·6190 (27)	0·1190 (12)	0·4345 (14)	1·0	63 (7)
C(11)	0·5567 (25)	0·0797 (13)	0·3663 (12)	1·0	58 (6)
C(12)	0·5455 (25)	0·1303 (11)	0·2935 (12)	1·0	46 (6)
C(13)	0·4900 (26)	0·1111 (10)	0·2103 (12)	1·0	52 (7)
C(131)	0·4925 (16)	0·0295 (7)	0·1847 (10)	1·0	53 (6)
C(132)	0·6104 (16)	−0·0164 (7)	0·2069 (10)	1·0	62 (7)
C(133)	0·6076 (16)	0·0934 (7)	0·1837 (10)	1·0	78 (8)
C(134)	0·4868 (16)	−0·1245 (7)	0·1381 (10)	1·0	78 (8)
C(135)	0·3688 (16)	−0·0786 (7)	0·1159 (10)	1·0	80 (8)
C(136)	0·3717 (16)	−0·0016 (7)	0·1391 (10)	1·0	63 (7)
N(2)	0·4130 (18)	0·2393 (9)	0·1650 (10)	1·0	42 (4)
C(14)	0·4457 (24)	0·1662 (10)	0·1485 (11)	1·0	55 (6)
C(15)	0·4198 (22)	0·1525 (12)	0·0573 (11)	1·0	48 (6)
C(16)	0·3630 (25)	0·2174 (11)	0·0196 (13)	1·0	57 (6)
C(17)	0·3571 (24)	0·2719 (11)	0·0893 (12)	1·0	55 (6)
C(18)	0·3021 (27)	0·3458 (11)	0·0799 (14)	1·0	50 (8)
C(181)	0·1992 (14)	0·3649 (9)	0·0039 (8)	1·0	44 (5)
C(182)	0·0885 (14)	0·3150 (9)	−0·0219 (8)	1·0	73 (8)
C(183)	−0·0063 (14)	0·3318 (9)	−0·0938 (8)	1·0	67 (7)
C(184)	0·0097 (14)	0·3985 (9)	−0·1400 (8)	1·0	70 (7)
C(185)	0·1204 (14)	0·4484 (9)	−0·1143 (8)	1·0	66 (7)
C(186)	0·2152 (14)	0·4316 (9)	−0·0423 (8)	1·0	71 (8)
N(3)	0·4448 (19)	0·4047 (10)	0·2027 (11)	1·0	56 (5)
C(19)	0·3301 (22)	0·4029 (12)	0·1411 (13)	1·0	53 (6)
C(20)	0·2602 (26)	0·4740 (12)	0·1519 (16)	1·0	70 (7)
C(21)	0·3282 (24)	0·5166 (14)	0·2143 (15)	1·0	71 (7)
C(22)	0·4470 (20)	0·4714 (11)	0·2485 (12)	1·0	53 (6)
C(23)	0·5502 (23)	0·4938 (10)	0·3158 (13)	1·0	62 (6)
C(231)	0·5682 (18)	0·5761 (7)	0·3395 (10)	1·0	59 (6)

Table 1 (*cont.*)

	x	y	z	k	$U_{\rm eq}/U$
C(232)	0·5669 (18)	0·6301 (7)	0·2745 (10)	1·0	71 (7)
C(233)	0·5859 (18)	0·7075 (7)	0·2946 (10)	1·0	102 (10)
C(234)	0·6061 (18)	0·7307 (7)	0·3796 (10)	1·0	93 (10)
C(235)	0·6074 (18)	0·6767 (7)	0·4446 (10)	1·0	81 (8)
C(236)	0·5884 (18)	0·5994 (7)	0·4245 (10)	1·0	64 (7)
N(4)	0·6103 (19)	0·3648 (9)	0·3727 (11)	1·0	54 (5)
C(24)	0·6378 (20)	0·4411 (10)	0·3642 (13)	1·0	46 (5)
C(25)	0·7711 (23)	0·4577 (13)	0·4145 (15)	1·0	66 (7)
C(26)	0·8164 (24)	0·3908 (11)	0·4518 (15)	1·0	60 (6)
C(27)	0·7174 (27)	0·3306 (11)	0·4264 (18)	1·0	59 (9)
C(28)	0·7190 (20)	0·2546 (10)	0·4525 (11)	1·0	44 (5)
C(281)	0·8082 (18)	0·2358 (11)	0·5352 (9)	1·0	67 (7)
C(282)	0·8034 (18)	0·2829 (11)	0·6059 (9)	1·0	82 (9)
C(283)	0·8848 (18)	0·2655 (11)	0·6830 (9)	1·0	110 (11)
C(284)	0·9710 (18)	0·2009 (11)	0·6892 (9)	1·0	102 (11)
C(285)	0·9758 (18)	0·1538 (11)	0·6185 (9)	1·0	102 (10)
C(286)	0·8944 (18)	0·1713 (11)	0·5414 (9)	1·0	80 (8)

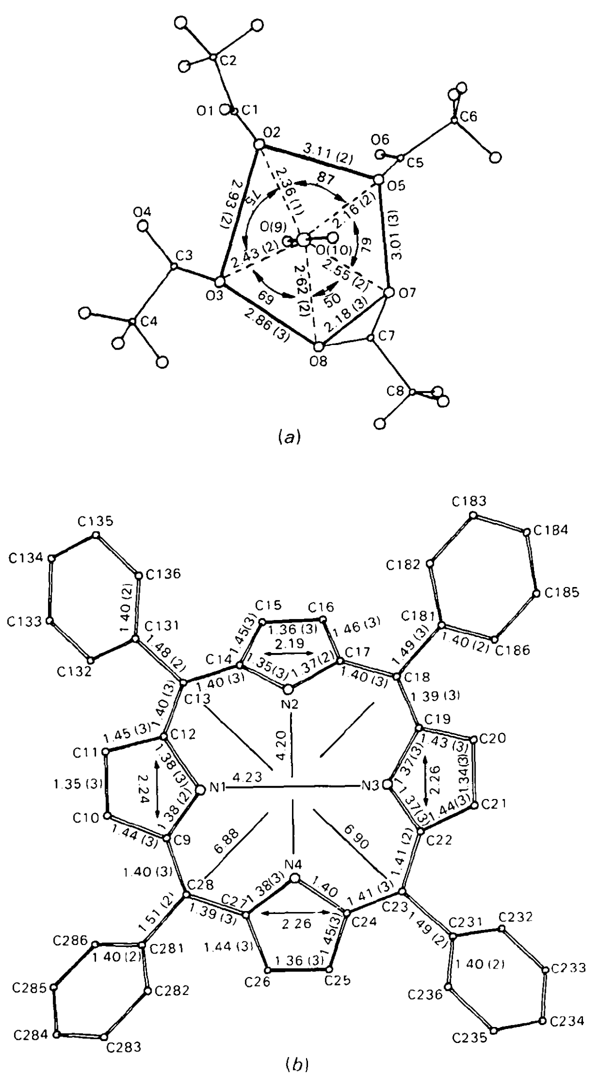

Fig. 2. (*a*) The coordination in $UO_2(tfa)_2(Htfa)_2$ (distances in Å, angles in deg) [U—O(9) = 1·70 (2), U—O(10) = 1·81 (2) Å]. (*b*) Distances (Å) in the TPP molecules.

Table 2. *Principal distances* (Å) *and angles* (°) *in the tfa groups*

O(1)—C(1)	1·20 (2)	O(3)—C(3)	1·25 (2)	O(5)—C(5)	1·26 (2)	O(7)—C(7)	1·19 (3)
O(2)—C(1)	1·28 (2)	O(4)—C(3)	1·20 (2)	O(6)—C(5)	1·25 (3)	O(8)—C(7)	1·19 (3)
C(1)—C(2)	1·55 (2)	C(3)—C(4)	1·55 (2)	C(5)—C(6)	1·50 (2)	C(7)—C(8)	1·55 (2)
O(1)—C(1)—O(2)	131 (1)	O(3)—C(3)—O(4)	131 (1)	O(5)—C(5)—O(6)	131 (2)	O(7)—C(7)—O(8)	132 (2)
O(1)—C(1)—C(2)	120 (1)	O(3)—C(3)—C(4)	114 (1)	O(5)—C(5)—C(6)	114 (1)	O(7)—C(7)—C(8)	114 (1)
O(2)—C(1)—C(2)	109 (1)	O(4)—C(3)—C(4)	115 (1)	O(6)—C(5)—C(6)	115 (1)	O(8)—C(7)—C(8)	114 (2)

to TPP; O(5) of a third monodentate tfa and finally the O(7) and O(8) of a bidentate tfa group, giving a polyhedron described as an irregular pentagonal bipyramid (Fig. 2*a*). The distances of these O and U atoms to the best least-squares equatorial plane are: +0·006 (U), −0·10 [O(2)], +0·07 [O(3)], −0·05 [O(8)], −0·03 [O(7)], and +0·04 Å [O(5)]. The uranyl bonds are perpendicular to the equatorial plane.

The interatomic distances in the two 'protonated tfa' groups are close to those of trifluoroacetic acid (Nahringbauer, Lundgren & Andersen, 1979). The H atoms of these molecules were not located. The two C—O distances in the third unidentate tfa group are not differentiated and the distance C(5)—C(6) is a little shorter than normal. In contrast, in the bidentate tfa group, the distance C(7)—C(8) is normal but the C—O distances are rather short. The trifluoromethyl groups occupy many positions. The mean distance C—F is 1·33 Å and the F—C—F angles take values between 106 and 113° for all positions. In addition, slightly high thermal parameters are found for O(6), the non-bonded O of the third monodentate tfa, and C(73) of bidentate tfa. The principal distances and angles of the tfa groups are summarized in Table 2.

From Fig. 2(*b*) and values of the angles in the TPP molecule it can be seen that the TPP in this crystal has very similar distances and angles to those of free tetragonal and triclinic TPP within the limits imposed by the accuracy (Silvers & Tulinsky, 1967; Hamor, Hamor & Hoard, 1963). A closer examination of the distribution and deviations from planarity of TPP reveals that TPP has approximate molecular symmetry, as required in tetragonal TPP (some deviations are indicated in Table 3).

The greatest difference between them is the tilt of the pyrrole and phenyl rings. The four pyrrole rings are planar [standard deviations for the distances to the best least-squares pyrrole planes: 0·003, 0·017, 0·006 and 0·003 Å for N(1), N(2), N(3) and N(4) pyrrole rings respectively] and are inclined 36, 24, 28 and 31° to the best plane through the four N atoms. Substituent aromatic rings are at 35, 35, 30 and 30° to this plane. The two inner-ring porphyrin H atoms are considered attached to the four central N atoms with an occupancy factor of one-half. This seems reasonable since the intramolecular differences in the pyrrole rings are small and also in view of the equivalent role of the N atoms in the crystal packing. This would imply rapid interconversion of N—H tautomers.

Table 3. *Deviations* (Å) *of atoms from the least-squares plane of the inner atoms in the TPP molecule*

N(1)	+0·08 (2)	N(3)	+0·09 (3)
C(12)	−0·22 (2)	C(22)	−0·23 (2)
C(13)	+0·04 (3)	C(23)	+0·06 (2)
C(14)	+0·30 (2)	C(24)	+0·29 (2)
N(2)	−0·04 (2)	N(4)	−0·11 (2)
C(17)	+0·22 (2)	C(27)	+0·26 (3)
C(18)	−0·07 (3)	C(28)	−0·06 (2)
C(19)	−0·32 (2)	C(9)	−0·32 (2)

References

Hamor, M. J., Hamor, T. A. & Hoard, J. L. (1963). *J. Am. Chem. Soc.* **86**, 1938–1942.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

Johnson, C. K. (1971). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J. P. & Woolfson, M. M. (1978). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain-la-Neuve, Belgium.

Nahringbauer, I., Lundgren, J. O. & Andersen, E. K. (1979). *Acta Cryst.* B**35**, 508–510.

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

Silvers, S. J. & Tulinsky, A. (1967). *J. Am. Chem. Soc.* **89**, 331–337.

Acta Cryst. (1983). C**39**, 1628–1630

The Structure of *catena*-Tribromo-μ-(1,4-dioxane-*O*,*O'*)-thallium(III), [$TlBr_3(C_4H_8O_2)$]

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Abstract. $M_r = 532{\cdot}1$, monoclinic, $C2/c$, $a = 11{\cdot}56$ (1), $b = 6{\cdot}966$ (5), $c = 15{\cdot}50$ (2) Å, $\beta = 122{\cdot}3$ (1)°, $V = 1054{\cdot}8$ Å^3, $Z = 4$, $D_x = 3{\cdot}35$, $D_m = 3{\cdot}288$ Mg m^{-3}, $\mu = 25{\cdot}9$ mm^{-1}, $F(000) = 936$, room temperature. The structure has been solved from 1110 diffractometer intensities with Mo $K\alpha$ radiation ($\lambda = 0{\cdot}7106$ Å) and

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