

monohydrate (Dhanaraj & Vijayan, 1987). Owing to different complications with the structures, accurate results are available only for a few of these determinations (the α form of choline chloride and choline meclofenamate monohydrate). The uranium(VI) complex has $R = 0.041$ (due to U), but the choline ion has high displacement parameters. Bond lengths and angles in the present structure are within the expected ranges.

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Acta Cryst. (1992). **C48**, 1345–1347

Structure of $[N,N,N',N'$ -Tetramethylguanidinium(1+) Pentachlorophenolate(1-) Pentachlorophenol]₂

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(Received 26 September 1991; accepted 3 December 1991)

Abstract. $(C_5H_{14}N_3^+ \cdot C_6Cl_5O^- \cdot C_6Cl_5OH)_2$, $M_r = 1295.71$, monoclinic, $P2_1/n$, $Z = 4$, $a = 15.603$ (3), $b = 13.7397$ (6), $c = 23.717$ (5) Å, $\beta = 90.280$ (15)°, $V = 5084.4$ (15) Å³, $D_x = 1.693$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 11.12$ cm⁻¹, $F(000) = 2592$, $R = 0.048$ for 6593 unique reflections with $I > 2.5\sigma(I)$. In the crystal structure the two tetramethylguanidine (TMG) fragments act as a 'proton sponge' by accepting a proton from two of the four pentachlorophenols in the asymmetric unit. The TMG fragments are planar and approach C_{3h} symmetry. With the exception of one N—H donor, all N—H and O—H hydrogen-bond donors are involved in intermolecular hydrogen bonds.

Experimental. Crystals of the title compound were obtained from a solution in acetonitrile of pentachlorophenol and N,N,N',N' -tetramethylguanidine in

a ratio of 2:1. A colourless crystal of dimensions 0.18 × 0.30 × 0.35 mm was used for data collection on an Enraf-Nonius CAD-4 diffractometer with Zr-filtered Mo $K\alpha$ radiation with the ω -2 θ scan mode, $\omega = [0.60 + 0.35 \tan \theta]^\circ$. Lattice parameters were determined from the setting angles of 25 reflections in the range $14.1 < \theta < 19.0^\circ$. Intensity data of 13 263 reflections were measured ($h - 20 \rightarrow 20$; $k 0 \rightarrow 18$; $l 0 \rightarrow 31$, $2\theta_{max} = 55^\circ$), 11 253 unique ($R_{int} = 0.034$) and 6593 above the $2.5\sigma(I)$ level.

Three periodically measured standard reflections (240, 204, 044) showed an average deviation of less than 1% during 190 h of X-ray exposure. Intensities were corrected for Lp effects. The structure was solved by direct methods with *SHELXS86* (Sheldrick, 1986). The methyl-group H atoms were placed at calculated positions (C—H 1.00 Å) and refined riding on their carrier atoms. The phenolic and amino-group H atoms were located in electron-density difference maps and kept fixed during

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refinement. Weighted anisotropic full-matrix least-squares refinement of 579 parameters on F of non-H atoms, H atoms isotropic with an overall temperature factor, converged at $R = 0.048$, $wR = 0.043$ and $w = [\sigma^2(F_o)]^{-1}$, $S = 2.06$, $(\Delta/\sigma)_{av} = 0.0196$, $(\Delta/\sigma)_{max} = 0.177$. An empirical extinction parameter refined to 2.8×10^{-4} . Absorption correction by the *DIFABS* procedure (Walker & Stuart, 1983), minimum and maximum transmission 0.719 and 1.209, respectively. Maximum and minimum residual densities in the final difference map 0.41 and $-0.39 \text{ e } \text{Å}^{-3}$, respectively. Scattering factors from Cromer & Mann (1968) and anomalous-dispersion terms from Cromer & Liberman (1970). Calculations were performed with *SHELX76* (Sheldrick, 1976) (refinement) and the *EUCLID* package (Spek, 1982) (geometry and illustrations) on a MicroVAX II computer. A view of the structure with atom numbering is shown in Fig. 1. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,* average bond

* Tables of observed and calculated structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54923 (43 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GE0296]

distances and angles of chemically similar fragments in Table 2 and hydrogen-bond parameters in Table 3.

Related literature. The structure represents a proton-transfer complex in which two of the four pentachlorophenol (PCP) residues each donate a proton to a guanidine residue.

Table 1. *Final atomic coordinates with e.s.d.'s in parentheses*

	$U_{eq} = (1/3)(\text{trace of orthogonalized } U_{ij} \text{ tensor}).$			
	x	y	z	$U_{eq}(\text{Å}^2)$
[TMG-1]⁺				
N(1)	0.2373 (2)	0.2499 (3)	0.1288 (1)	0.047 (1)
N(2)	0.3144 (2)	0.1053 (2)	0.1220 (1)	0.043 (1)
N(3)	0.3688 (2)	0.2344 (2)	0.1727 (1)	0.044 (1)
C(25)	0.3072 (3)	0.1964 (3)	0.1412 (2)	0.038 (1)
C(26)	0.2136 (3)	0.3329 (3)	0.1631 (2)	0.061 (2)
C(27)	0.1964 (3)	0.2470 (4)	0.0734 (2)	0.077 (2)
C(28)	0.2405 (3)	0.0422 (3)	0.1121 (2)	0.071 (2)
C(29)	0.3970 (3)	0.0548 (3)	0.1225 (2)	0.053 (2)
[TMG-2]⁺				
N(4)	0.6782 (2)	0.3381 (3)	0.1344 (1)	0.046 (1)
N(5)	0.8229 (2)	0.3168 (3)	0.1140 (1)	0.052 (1)
N(6)	0.7326 (3)	0.1850 (3)	0.1203 (2)	0.073 (2)
C(30)	0.7436 (3)	0.2809 (3)	0.1232 (2)	0.045 (2)
C(31)	0.8981 (3)	0.2537 (4)	0.1183 (2)	0.083 (2)
C(32)	0.8354 (4)	0.4074 (4)	0.0839 (2)	0.088 (3)
C(33)	0.6870 (3)	0.4312 (3)	0.1629 (2)	0.077 (2)
C(34)	0.5895 (3)	0.3000 (4)	0.1315 (2)	0.069 (2)
[PCP-1]				
Cl(1)	0.35842 (7)	0.33266 (8)	0.30946 (5)	0.0517 (4)
Cl(2)	0.36128 (7)	0.31884 (9)	0.44035 (5)	0.0573 (4)
Cl(3)	0.52496 (8)	0.38071 (9)	0.50754 (4)	0.0569 (4)
Cl(4)	0.68074 (7)	0.46641 (9)	0.44287 (5)	0.0615 (5)
Cl(5)	0.67415 (8)	0.4865 (1)	0.31215 (5)	0.0653 (5)
O(1)	0.5188 (2)	0.4175 (2)	0.2608 (1)	0.053 (1)
C(1)	0.5152 (3)	0.4103 (3)	0.3169 (2)	0.039 (1)
C(2)	0.4464 (2)	0.3725 (3)	0.3467 (2)	0.037 (1)
C(3)	0.4488 (3)	0.3644 (3)	0.4050 (2)	0.038 (1)
C(4)	0.5207 (3)	0.3925 (3)	0.4353 (2)	0.040 (1)
C(5)	0.5905 (3)	0.4301 (3)	0.4063 (2)	0.041 (1)
C(6)	0.5884 (3)	0.4394 (3)	0.3479 (2)	0.041 (1)
[PCP-2]⁻				
Cl(6)	0.90113 (9)	0.41076 (9)	0.24311 (5)	0.0686 (5)
Cl(7)	0.90374 (8)	0.19437 (9)	0.28420 (5)	0.0630 (4)
Cl(8)	0.90831 (8)	0.14868 (8)	0.41326 (5)	0.0624 (5)
Cl(9)	0.90298 (9)	0.3182 (1)	0.49897 (5)	0.0721 (5)
Cl(10)	0.89713 (8)	0.53313 (8)	0.45738 (5)	0.0566 (4)
O(2)	0.8950 (2)	0.5545 (2)	0.3357 (1)	0.049 (1)
C(7)	0.8998 (2)	0.4621 (3)	0.3522 (2)	0.041 (2)
C(8)	0.9017 (3)	0.3845 (3)	0.3139 (2)	0.043 (1)
C(9)	0.9036 (2)	0.2883 (3)	0.3322 (2)	0.043 (2)
C(10)	0.9043 (2)	0.2674 (3)	0.3893 (2)	0.043 (2)
C(11)	0.9024 (3)	0.3432 (3)	0.4280 (2)	0.043 (1)
C(12)	0.9000 (2)	0.4390 (3)	0.4097 (2)	0.041 (1)
[PCP-3]				
Cl(11)	0.35047 (7)	0.06022 (9)	0.28557 (5)	0.0604 (4)
Cl(12)	0.36279 (9)	0.0552 (1)	0.41719 (5)	0.0694 (5)
Cl(13)	0.53202 (9)	0.12079 (9)	0.47699 (5)	0.0683 (5)
Cl(14)	0.68512 (8)	0.1936 (1)	0.40538 (5)	0.0692 (5)
Cl(15)	0.67128 (8)	0.1972 (1)	0.27440 (5)	0.0717 (5)
O(3)	0.5047 (2)	0.1276 (2)	0.2304 (1)	0.047 (1)
C(13)	0.5117 (3)	0.1274 (3)	0.2858 (2)	0.040 (1)
C(14)	0.4432 (3)	0.0967 (3)	0.3195 (2)	0.040 (1)
C(15)	0.4486 (3)	0.0950 (3)	0.3777 (2)	0.042 (1)
C(16)	0.5230 (3)	0.1254 (3)	0.4047 (2)	0.044 (1)
C(17)	0.5914 (3)	0.1567 (3)	0.3725 (2)	0.043 (2)
C(18)	0.5863 (3)	0.1578 (3)	0.3142 (2)	0.042 (1)

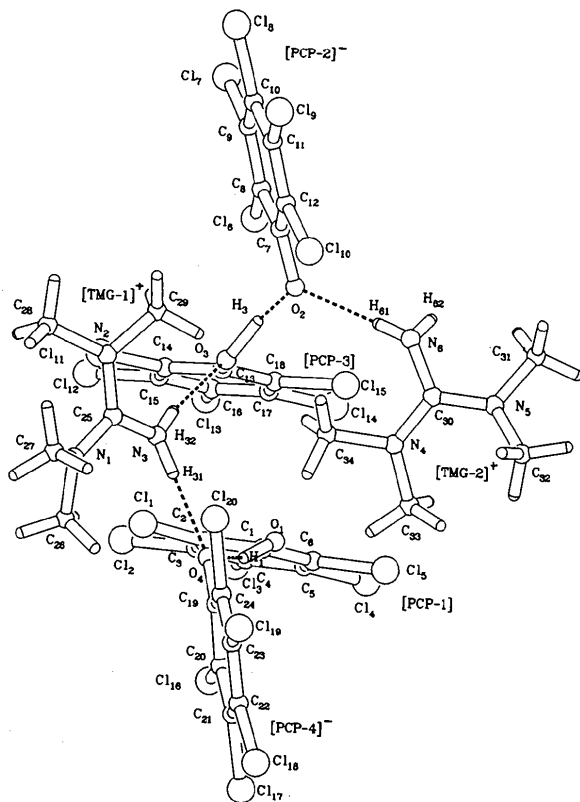


Fig. 1. View of the molecular structure with atom numbering. Hydrogen bonds are indicated by dashed lines.

Table 1 (*cont.*)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}(\text{\AA}^2)$
[PCP-4] ⁻				
Cl(16)	0.11676 (9)	0.10898 (8)	0.23817 (5)	0.0609 (4)
Cl(17)	0.12842 (8)	0.30347 (8)	0.30550 (6)	0.0647 (4)
Cl(18)	0.13633 (9)	0.29959 (9)	0.43688 (6)	0.0731 (5)
Cl(19)	0.13111 (1)	0.1008 (1)	0.49934 (5)	0.0798 (5)
Cl(20)	0.10927 (8)	-0.09147 (8)	0.43240 (5)	0.0596 (4)
O(4)	0.0981 (2)	-0.0645 (2)	0.3098 (1)	0.046 (1)
C(19)	0.1092 (2)	0.0161 (3)	0.3377 (2)	0.035 (1)
C(20)	0.1166 (3)	0.1069 (3)	0.3109 (2)	0.039 (1)
C(21)	0.1252 (3)	0.1933 (3)	0.3409 (2)	0.042 (1)
C(22)	0.1293 (3)	0.1926 (3)	0.3990 (2)	0.044 (2)
C(23)	0.1256 (3)	0.1037 (3)	0.4267 (2)	0.046 (2)
C(24)	0.1153 (3)	0.0179 (3)	0.3967 (2)	0.039 (1)

Table 2. Average bond distances (Å) and bond angles (°) of chemically similar fragments

Number of contributors		
[TMG-1] ⁺ and [TMG-2] ⁺		
8	N—C(<i>sp</i> ³)	1.460 (2)
6	N—C(<i>sp</i> ²)	1.333 (2)
4	C(<i>sp</i> ³)—N—C(<i>sp</i> ³)	114.8 (2)
8	C(<i>sp</i> ³)—N—C(<i>sp</i> ²)	121.6 (1)
6	N—C(<i>sp</i> ²)—N	120.0 (2)
[PCP-1] and [PCP-3]		
2	C—O	1.326 (4)
12	C—C	1.392 (2)
10	C—Cl	1.722 (2)
4	C—C—O	121.4 (2)
12	C—C—C	119.8 (1)
20	C—C—Cl	119.7 (1)
[PCP-2] ⁻ and [PCP-4] ⁻		
2	C—O	1.316 (4)
12	C—C	1.392 (2)
10	C—Cl	1.724 (1)
4	C—C—O	121.9 (2)
12	C—C—C	120.0 (1)
20	C—C—Cl	119.6 (1)

Table 3. Hydrogen-bond geometry (Å, °)

	D—H...A	D...A	D—H	H...A	D—H...A
O(1)—H(1)...O(4) ⁱ	2.482 (4)	0.93 (4)	1.63 (4)	149 (3)	
O(3)—H(3)...O(2) ⁱⁱ	2.437 (4)	1.14 (4)	1.32 (4)	166 (3)	
N(3)—H(31)...O(4) ⁱⁱ	2.841 (4)	0.89 (4)	1.97 (4)	168 (3)	
N(3)—H(32)...O(3) ⁱⁱⁱ	2.914 (4)	0.87 (4)	2.06 (4)	170 (4)	
N(6)—H(61)...O(2) ⁱⁱ	2.879 (5)	0.85 (6)	2.04 (5)	172 (5)	
Symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$; (iii) <i>x, y, z</i> .					

A similar proton-transfer complex involving bis-(dimethylamino)naphthalene (DMAN) and PCP, (DMANH⁺.PCP⁻.[PCP]₂), has recently been reported (Kanters, ter Horst, Kroon & Grech, 1992). However, in the structures of the complexes 4-methylpyridine.PCP (Malarski, Majerz & Lis, 1987*a*; Majerz, Malarski & Lis, 1990) and 3-pyridinecarboxitrile.PCP (Malarski, Majerz & Lis, 1987*b*) owing to the lower basicity of the pyridine constituent no proton transfer occurs.

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