H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.0904P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.004$
Absolute structure: Flack (1983)
Flack parameter = 0.11 (18)

Table 1. *Selected geometric parameters (Å, °)*

O1—C1	1.268 (6)	N1—C22	1.450 (8)
O2—C12	1.285 (7)	N2—C12	1.241 (6)
N1—C1	1.202 (7)	N2—C11	1.453 (9)
C1—N1—C22	124.7 (6)	N2—C11—C10	113.0 (7)
C12—N2—C11	120.8 (6)	N2—C12—O2	123.6 (6)
N1—C1—O1	123.2 (6)	N2—C12—C13	118.5 (6)
N1—C1—C2	122.4 (6)	O2—C12—C13	117.8 (5)
O1—C1—C2	114.2 (6)	N1—C22—C21	108.0 (6)
C22—N1—C1—O1	−0.9 (11)	C11—N2—C12—O2	2.6 (10)
N1—C1—C2—C3	97.3 (8)	N2—C12—C13—C14	106.3 (7)
O1—C1—C2—C3	−77.1 (8)	O2—C12—C13—C14	−70.5 (7)
C1—C2—C3—C4	−74.4 (8)	C12—C13—C14—C15	−73.6 (7)
C12—N2—C11—C10	108.8 (8)	C1—N1—C22—C21	109.4 (8)
C9—C10—C11—N2	−72.2 (8)	C20—C21—C22—N1	−69.2 (7)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···Cl$^{\text{i}}$	0.862	2.262	3.116 (6)	170.7
N2—H2N···Cl$^{\text{ii}}$	0.858	2.215	3.071 (5)	175.4
O2—H2O···O2$^{\text{iii}}$	1.206 (4)	1.206 (4)	2.406 (6)	171.9 (3)
O1—H1O···O1$^{\text{iii}}$	1.232 (4)	1.232 (4)	2.464 (6)	180.0 (6)

Symmetry codes: (i) $\frac{1}{4}+x, \frac{1}{4}-y, \frac{1}{4}+z$; (ii) $1+x, -y, -z$; (iii) $x, -y, -z$.

Two octants of reflections were measured in order to ameliorate the collected intensity values and they were sorted and merged before the structure solution and refinement.

All non-H atoms were refined anisotropically. The hydroxy-H atoms were located on the twofold axis in the middle of the hydrogen bonds linking two molecules. Attempts to define and refine disordered H-atom positions at the more usual O—H distance of 0.84 Å failed with the H atoms always returning to the twofold axis. This result is supported by the data in the Cambridge Structural Database (Allen & Kennard, 1993), where some 114 O—H distances are close to 1.23 Å when the O···O hydrogen-bonding distance is less than 2.5 Å (from 139 observations). It occurs, for example, in glyoximato or dihydrogenphosphato ions, where disorder of the H atom gives a symmetric hydrogen bond with a very long O—H distance. The positions of the (N)H and (C)H atoms were computed geometrically and refined using a riding model. An overall isotropic temperature factor was assumed for all H atoms. The polar-axis direction has been chosen arbitrarily as the standard uncertainty in the Flack parameter makes the result inconclusive.

Data collection: *CAD-4/PC* (Kretschmar, 1996). Cell refinement: *CAD-4/PC*. Data reduction: *CFEO* (Solans, 1978). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEP*3.2 (Brueggemann & Schmid, 1990). Software used to prepare material for publication: *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1071). Services for accessing these data are described at the back of the journal.

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3-Triphenylphosphoniopropionate dihydrate

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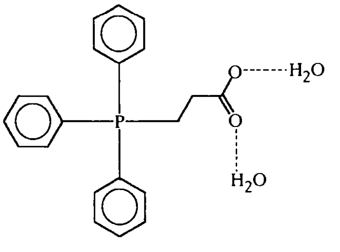
Abstract

Zwitterionic 3-triphenylphosphoniopropionate crystallizes as a dihydrate, $C_{21}H_{19}O_2P \cdot 2H_2O$, in which the carboxyl-O atoms are each hydrogen-bonded to a water molecule.

Comment

Betaine, $[(CH_3)_3NCH_2CO_2]$, and its analogs react with copper salts to furnish copper complexes that are rich in carboxylate ligands (Chen & Mak, 1994). Copper perchlorate forms tetrakisbetainecopper perchlorate with betaine (Ng *et al.*, 1998); the pyridiniopropionate homolog (Chen & Mak, 1992) reacts with lanthanum perchlorate to yield novel octadecanuclear copper(II)–lanthanoid(III) clusters (Chen *et al.*, 1996). The tertiary phosphine betaines, which can be synthesized by the carboxyalkylation of tertiary phosphines, are the phosphorus analogs of these tertiary amine betaines. Furthermore, owing to the presence of four P—C bonds, such compounds are expected to display phase-transfer catalytic properties. For the $(C_6H_5)_3P(CH_2)_nCO_2$ series, the $n = 1$ derivative is unstable (Denny & Smith, 1962), and the limited structural studies have concentrated on the stable $n = 2$ derivative, $[(C_6H_5)_3PCH_2CH_2CO_2]$. The compound has been characterized as in the ruthenium (Irvine *et al.*, 1991), organotin (Ng & Kumar Das, 1993), cadmium (Li & Mak, 1995) and mercury (Li & Mak, 1997) complexes. In the ruthenium and mercury complexes, the ligand behaves as a chelating entity whereas it behaves as a monodentate oxygen donor in the organotin complex. The ligand functions as a carboxylate bridge to link two metal atoms in the cadmium complexes. Our attempt to prepare the analogous copper–cerium cluster gave instead a cation whose two Cu and two Ce atoms are bonded to some eight ligands and ten water molecules (Ng, 1998).



(I)

This phosphobetaine, (I), exists as a dihydrate: each of the carboxyl-O atoms is hydrogen-bonded to a water molecule [O1···O3 2.515 (11) and O2···O4 2.698 (8) Å]; the water molecules are also linked together by hydrogen bonds [O3···O3^{i} 2.63 (2) Å; O3—O4^{ii} 2.501 (14) Å [symmetry codes: (i) $-1 - x$, $1 - y$, $1 - z$; (ii) $-x$, $1 - y$, $1 - z$]. The hydrogen bonds link the dihydrate into a one-dimensional chain. The carboxyl–water interactions open up the C—O angle to 126.1 (6)° relative to the idealized sp^2 angle of 120°. The phosphorus atom shows tetrahedral coordination. The packing is not compact as there are two voids of 37.2 Å^3 $(0,0,\frac{1}{2};\ \frac{1}{2},\frac{1}{2},0)$ in the crystal structure, as calculated by *PLATON* (Spek, 1990).

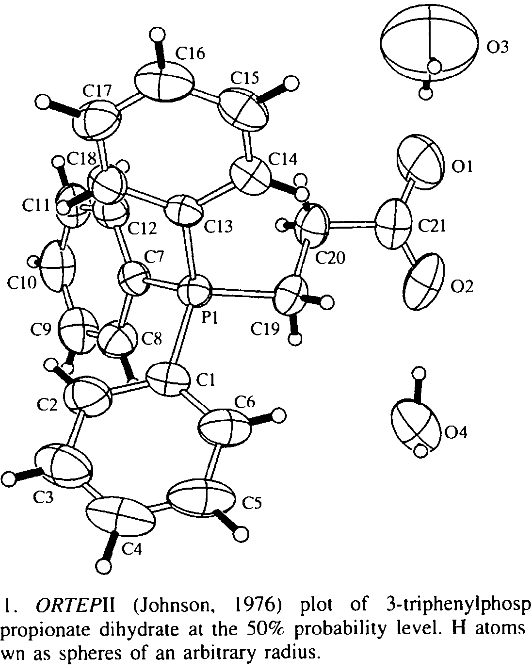


Fig. 1. *ORTEP*II (Johnson, 1976) plot of 3-triphenylphosphoniopropionate dihydrate at the 50% probability level. H atoms are drawn as spheres of an arbitrary radius.

Experimental

The phosphobetaine was synthesized by neutralizing triphenylphosphonium-3-propionic acid chloride dissolved in ethanol with aqueous sodium bicarbonate (Denny & Smith, 1962). The product, when recrystallized from 95% ethanol, gave the dihydrate. The anhydrous compound can be synthesized from the reaction of triphenylphosphine with acrylic acid (Tsivunin *et al.*, 1973).

Crystal data

$C_{21}H_{19}O_2P{\cdot}2H_2O$
$M_r = 370.36$
Monoclinic
$P2_1/n$
$a = 9.1250\ (7)$ Å
$b = 12.769\ (1)$ Å
$c = 16.428\ (2)$ Å
$\beta = 98.738\ (9)°$
$V = 1892.0\ (3)$ Å^3
$Z = 4$
$D_x = 1.300$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8.0$–$10.0°$
$\mu = 0.168$ mm^{-1}
$T = 298\ (2)$ K
Block
0.36 × 0.36 × 0.18 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scans (North *et al.*, 1968)
$T_{\min} = 0.897$, $T_{\max} = 0.935$
3547 measured reflections
3325 independent reflections

1950 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.026$
$\theta_{\max} = 24.98°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 15$
$l = -19 \rightarrow 19$
3 standard reflections frequency: 60 min intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.073$
$wR(F^2) = 0.215$
$S = 1.019$
3325 reflections
235 parameters
H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.1116P)^2 + 0.5426P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.275$ e Å^{-3}
$\Delta\rho_{\min} = -0.517$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

P1—C1	1.796 (4)	P1—C19	1.795 (4)
P1—C7	1.794 (4)	O1—C21	1.239 (7)
P1—C13	1.789 (4)	O2—C21	1.210 (7)
C1—P1—C7	108.9 (2)	C7—P1—C19	109.4 (2)
C1—P1—C13	107.1 (2)	O1—C21—O2	126.1 (6)
C1—P1—C19	110.8 (2)	O1—C21—C20	116.1 (5)
C7—P1—C13	110.8 (2)	O2—C21—C20	117.8 (6)

H atoms bonded to C were treated using a riding model with $U(\mathrm{H}) = 1.5U_{\mathrm{eq}}(\mathrm{C})$. Water H atoms were placed at calculated positions with $U = 0.05$ Å^2. For the O3 water molecule, one of the H atoms is necessarily disordered over two positions.

Data collection: *CAD-4/PC* (Kretschmar, 1994). Cell refinement: *CELDIM* (Enraf–Nonius, 1988). Data reduction: *XCAD*4 (Harms, 1997). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*b*). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*97.

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Butyldichloro(phenylethynyl)tellurium(IV)

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Abstract

In the title compound, $[\mathrm{TeCl_2(C_4H_9)(C_8H_5)}]$ ($\mathrm{C_{12}H_{14}Cl_2Te}$), the intramolecular geometry around the $\mathrm{Te^{IV}}$ is pseudo-trigonal bipyramidal, with the Cl atoms in the apical positions and the Te lone pair occupying the fifth position in the equatorial plane. Taking into account the intermolecular secondary Te···Cl1 contact of 3.266 (1) Å, *trans* to the Te—$\mathrm{C_{vinyl}}$, the structure may be regarded as pseudo-octahedral. Distances and angles are: Te—Cl 2.486 (1) and 2.551 (1), Te—C 2.020 (3) and 2.146 (3) Å; Cl—Te—Cl 173.81 (3), Cl—Te—C 89.94 (10), 86.59 (9), 87.84 (10) and 87.98 (9), and C—Te—C 97.66 (13)°. All of the C atoms lie in a plane, with the Te atom displaced 0.2220 (2) Å from the plane in the direction of Cl2.

Comment

Butyltelluroalkynes are important intermediates in organic synthesis, since they permit the stereospecific synthesis of vinylic tellurium species (Comasseto *et al.*, 1997), which in turn are precursors of other vinyl metal derivatives such as vinyl lithium, copper, zinc, magnesium, sodium and calcium. In previous papers, we reported the synthesis (Dabdoub *et al.*, 1988) and reactions (Dabdoub & Comasseto, 1988; Dabdoub & Cassol, 1995) of these acetylenic tellurium species. Reaction of acetylenic tellurides with Br or I produces organyltellurium trihalides and the corresponding 1,1,2-trihaloethylenes, instead of the expected diorganyl tellurium dihalides (Dabdoub *et al.*, 1990). However, reaction of telluroacetylenes with an excess of sulfurylchloride in petroleum ether at 273 K affords the corresponding dichlorotelluroacetylenes in high yield. In this way, the phenylethynyl(butyltelluro) dichloride was obtained in 97% yield. Due to our interest in the structure of organotellurium compounds in general (Zukerman-Schpector *et al.*, 1995; Zukerman-Schpector, Cara-