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Crystallographic report

1,4-Bis(carboxymethyldiphenylphosphonio)butane dinitrate, $(CH_2)_4[(HOOCCH_2)Ph_2P^{(+)}]_2\cdot 2(NO_3^{(-)})$

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The centrosymmetric $\{[(HOOCCH_2PPh_2)]_2(CH_2)_4\}^{2+}$ cation adopts an extended conformation in which the phosphorus center adopts a tetrahedral geometry. O–H···O and C–H···O hydrogen bonding interactions expand this structure to form a two-dimensional layered architecture. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; phosphine betaines; hydrogen bond

COMMENT

Recently, we have reported the crystal structure of a protonated double phosphine betaine 1,4-bis(carboxymethyldiphenylphosphonio)butane dibromide (**1**), which is expected to have phase-transfer catalytic properties owing to the presence of four P–C bonds.[1] The structure of its nitrate analog, 1,4-bis(carboxymethyldiphenylphosphonio)butane dinitrate (**2**) comprises a centrosymmetric and doubly protonated phosphobetaine cation (Fig. 1) and two nitrate anions with tetrahedral phosphorus. An interesting structural feature of **2** resides in the formation of a two-dimensional hydrogen-bonded sheet. Intermolecular C10–H10A···O2^{i} hydrogen bonds ($i = -x+1, -y, -z$) link the cations into a chain. These helices are further extended to a two-dimensional layered architecture through O1–H1···O5, C2–H2B···O3^{ii} ($ii = -x+1.5, 0.5+y, 0.5-z$) and C15–H15A···O5^{iii} ($iii = x, 1+y, z$) hydrogen bonds between the cations and anions.

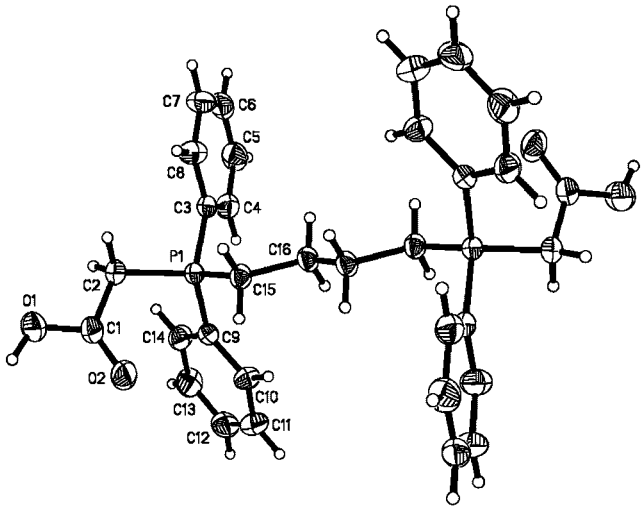

Figure 1. ORTEP diagram of the cation in **2** (NO_3^- anions are omitted for clarity). Key geometric parameters: P1–C2 1.804(3), P1–C3 1.788(3), P1–C9 1.786(3), P1–C15 1.797(3), O1–C1 1.291(3), O2–C1 1.205(3) Å; C2–P1–C3 105.65(12), C2–P1–C9 109.99(13), C2–P1–C15 108.74(13), C3–P1–C9 110.82(13), C3–P1–C15 110.17(12), C9–P1–C15 111.30(12)°.

EXPERIMENTAL

Zwitterionic 1,4-bis(carboxymethyldiphenylphosphonio)butane was prepared according to the literature method.[2] Single crystals of **2** were obtained by recrystallizing the above phosphine betaine from an H_2O–C_2H_5OH mixture in the presence of several drops of HNO_3. Intensity data for **2** were collected at 293(2) K on a Bruker Smart 1000 CCD diffractometer on a colorless block with dimensions of $0.25 \times 0.28 \times 0.34$ mm^3. $C_{32}H_{34}N_2O_{10}P_2$, $M = 668.55$, monoclinic, $P2_1/n$, $a = 14.051(4)$, $b = 7.613(2)$, $c = 15.419(5)$ Å, $\beta = 100.959(7)°$, $V = 1619.4(9)$ Å^3, $Z = 2$, 3902 unique data ($\theta_{max} = 28.1°$), 2423 data with $I \geq 2\sigma(I)$, $R = 0.059$ (obs.), $wR = 0.181$ (all data). Programs used: SMART,

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SAINT, SHELXL97 and SHELXTL. CCDC deposition number: 239446.

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