APPLIED ORGANOMETALLIC CHEMISTRY
Appl. Organometal. Chem. 2004; **18**: 499–500
Published online in Wiley InterScience (www.interscience.wiley.com). DOI:10.1002/aoc.713

Crystallographic report

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

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Received 6 May 2004; Revised 17 May 2004; Accepted 19 May 2004

The centrosymmetric cation $\{[(HOOCCH_2PPh_2)]_2(CH_2)_4\}^{2+}$ adopts an extended conformation. The phosphorus atom shows a tetrahedral coordination and each O–H of the carboxylic group is hydrogen bonded to a bromide ion. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; phosphine betaines; hydrogen bond

COMMENT

Tertiary phosphine betaines are expected to display phase-transfer catalytic properties owing to the presence of four P–C bonds. Recently, the crystal structure of 3-triphenylphosphoniopropionate dihydrate was reported.[1] Here, we describe the structure of a protonated double phosphine betaine, namely 1,4-bis(carboxymethyldiphenylphosphonio)butane dibromide, $(CH_2)_4[(HOOCCH_2)Ph_2P^{(+)}]_2{\cdot}2Br^{(-)}$ (**1**). The structure of **1** comprises a doubly protonated phosphobetaine cation (Fig. 1) and two bromide anions. The cationic unit is centrosymmetric and each phosphorus center shows a tetrahedral environment defined by four carbon atoms. An analysis of the crystal packing shows the existence of an O(1)–H(1)···Br(1)^i hydrogen bond ($i = 3/2 - x, -1/2 + y, 3/2 - z$); the O···Br separation is 3.133(4) Å with the H···Br distance being 2.23 Å and the angle at the hydrogen being 164°.

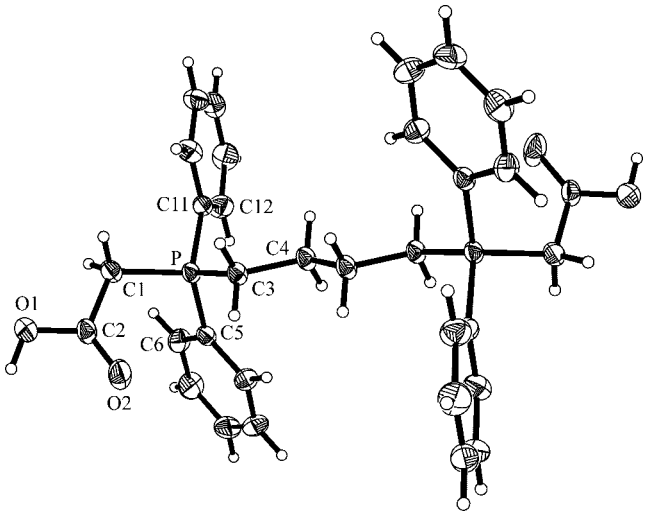

Figure 1. ORTEP diagram of **1** (Br^- anions are omitted for clarity). Key geometric parameters: P–C1 1.802(5), P–C3 1.797(5), P–C5 1.795(5), P–C11 1.796(5), O1–C2 1.293(6), O2–C2 1.187(6) Å; C1–P–C3 108.2(2), C1–P–C5 110.7(2), C1–P–C11 105.0(2), C3–P–C5 111.2(2), C3–P–C11 110.9(2), C5–P–C11 110.7(2)°.

EXPERIMENTAL

1,4-Bis(carboxymethyldiphenylphosphonio)butanedibromide was prepared according to the literature method.[2] Single crystals of **1** were obtained by recrystallizing the sample from an H_2O–C_2H_5OH mixture in the presence of several drops of HBr. Intensity data for **1** were collected at 293(2) K on a Bruker Smart 1000 CCD diffractometer on a colorless block with dimensions of 0.40 × 0.42 × 0.42 mm^3. $C_{32}H_{34}Br_2O_4P_2$, $M = 704.33$, monoclinic, $P2_1/n$, $a = 14.4832(7)$, $b = 7.4772(4)$, $c = 14.8990(7)$ Å, $\beta = 103.696(1)°$, $V = 1567.59(14)$ Å^3, $Z = 2$, 2755 unique data ($\theta_{max} = 25.0°$), 2028 data with $I \geq 2\sigma(I)$, $R = 0.057$ (obs.), $wR = 0.148$ (all data). Programs used: SMART, SAINT, SHELXL97, and SHELXTL. CCDC deposition number: 237669.

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Contract/grant sponsor: Starting Funding of Tianjin Normal University.
Contract/grant sponsor: Natural Science Foundation of Tianjin; Contract/grant number: 033609711.

Acknowledgements

This work was supported financially by the Starting Funding of Tianjin Normal University and the Natural Science Foundation of Tianjin (033609711).

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