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Crystallographic report

Dimethylammonium phenylphosphonate·2(phenylphosphonic acid)

Jens Beckmann*, Dainis Dakternieks and Andrew Duthie

Centre for Chiral and Molecular Technologies, Deakin University, Geelong 3217, Australia

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Individual molecules of $NH_2Me_2(HO)O_2PPh{\cdot}2PhPO(OH)_2$ are associated by hydrogen bonding, giving rise to a three-dimensional supramolecular array. Copyright © 2003 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; hydrogen bonding; supramolecular chemistry

COMMENT

Co-crystals of $H_2NMe_2(HO)O_2PPh{\cdot}2PhPO(OH)_2$ were isolated from a reaction mixture comprising $PhPO(OH)_2$ and $Me_2Si(NMe_2)_2$ in an attempt to prepare polymers

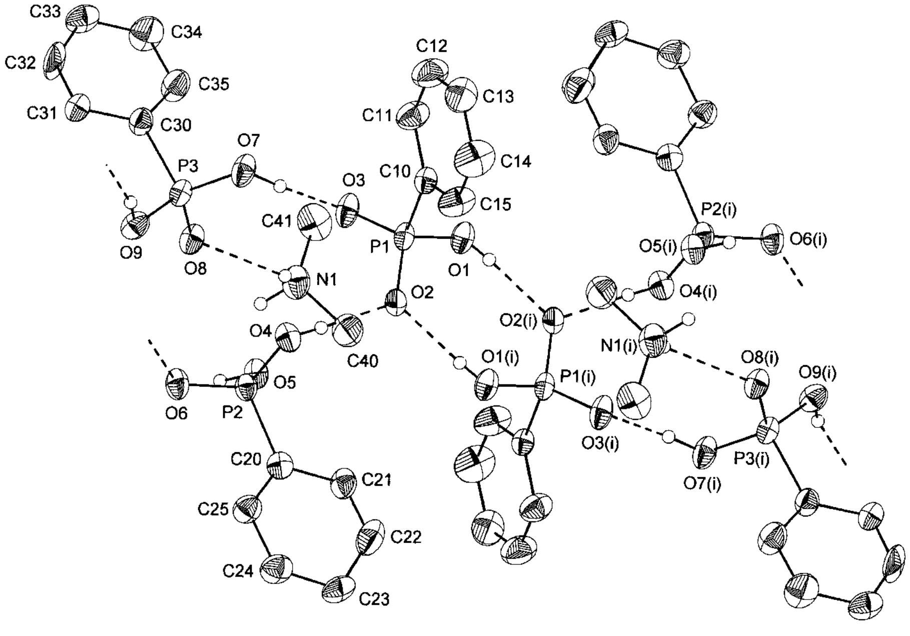

Figure 1. Molecular aggregation in $NH_2Me_2(HO)O_2PPh{\cdot}2PhPO(OH)_2$; hydrogen atoms attached to carbon atoms are omitted. Selected geometric parameters: P1–O1 1.562(2), P1–O2 1.504(1), P1–O3 1.498(2), P1–C10 1.790(2), P2–O4 1.543(2), P2–O5 1.541(2), P2–O6 1.485(2), P2–C20 1.786(3), P3–O7 1.530(2), P3–O8 1.490(2), P3–O9 1.545(2), P3–C30 1.780(3) Å; O1–P1–O2 109.48(8), O1–P1–O3 106.32(7), O2–P1–O3 116.17(8), O1–P1–C10 107.06(7), O2–P1–C10 108.01(8), O3–P1–C10 109.46(7), O4–P2–O5 106.67(7), O4–P2–O6 110.73(7), O5–P2–O6 113.99(7), O4–P2–C20 106.53(8), O5–P2–C20 107.25(8), O6–P2–C20 111.28(8), O7–P3–O8 113.27(7), O7–P3–O9 110.27(7), O8–P3–O9 108.80(7), O7–P3–C30 106.81(8), O8–P3–C30 110.48(8), O9–P3–C30107.29(8)°. Hydrogen bonding contacts: O2···O4 2.523(3), O3···O7 2.451(3), O6···O9(i) 2.535(3), N1···O8 2.830(4), O1···O2(i) 2.621(4) Å. Symmetry operation, (i): $-x$, $-y$, $-z$.

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*Correspondence to: Jens Beckmann, Centre for Chiral and Molecular Technologies, Deakin University, Geelong 3217, Australia.
E-mail: beckmann@deakin.edu.au
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with Si–O–P linkages. The four individual molecules, i.e. $H_2NMe_2^+$, $PhPO_2(OH)^-$ and the two $PhPO(OH)_2$, are associated by a variety of hydrogen bonds (Fig. 1). Although the crystal structure of $PhPO(OH)_2$ has been known since 1976,[1] there appears to be some current interest in supramolecular aspects of organophosphonic acids and related amine complexes.[2,3]

EXPERIMENTAL

A mixture of $PhPO(OH)_2$ (0.50 g, 3.16 mmol) and $Me_2Si(NMe_2)_2$ (0.46 g, 3.16 mmol) in dry toluene (50 ml) was stirred at room temperature overnight. The solvent was removed and 1 : 1 hexane/dichloromethane (20 ml) added. After thorough stirring the mixture was filtered. A few single crystals (m.p. 63–65 °C) suitable for X-ray crystallography were obtained by slow evaporation of the solvent from the filtrate. Intensity data were collected at 293 K on a Bruker SMART Apex CCD diffractometer for a block $0.40 \times 0.40 \times 0.40$ mm^3. $C_{20}H_{28}NO_9P_3$, $M = 519.34$, triclinic, $P\overline{1}$, $a = 7.9650(9)$, $b = 11.9830(13)$, $c = 12.9204(14)$ Å, $\alpha = 91.235(2)°$ $\beta = 96.661(2)°$, $\gamma = 98.700(2)°$, $V = 1209.8(2)$ Å^3, $Z = 2$, 5453 unique data ($\theta_{max} = 27.7°$), $R = 0.038$ (4818 [$I > 2\sigma(I)$] reflections), $wR = 0.113$ (all data), $\rho_{max} = 0.31$ e Å^{-3}. Disorder was resolved for the phenyl groups of the $PhPO(OH)_2$ molecules so that C21–C25 and C31–C35 were each refined over two sites with occupancy factors of 50 : 50. Programs used: SAINT, SADABS, SHELX-97. CCDC deposition number: 208288.

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