

10-Methoxycarbonyldecyltriphenylphosphonium Iodide

BY JILL C. VAN NIEKERK AND LUIGI R. NASSIMBENI*

Department of Physical Chemistry, University of Cape Town, South Africa

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Abstract. $C_{30}H_{38}O_2P^+I^-$, monoclinic, $P2_1/c$, $a = 9.540$ (5), $b = 16.670$ (8), $c = 18.570$ (9) Å, $\beta = 94.95$ (5)°, $M_r = 639.8$, $Z = 4$. The structure was determined by Patterson and Fourier methods and refined by full-matrix least squares to a final R of 0.046 for 2514 unique reflexions. The long aliphatic chain is in the extended configuration.

Introduction. The compound was prepared by reacting triphenylphosphine with methyl 11-iodohendecanoate and recrystallized from chloroform–methanol. It was required as an intermediate in the synthesis of Δ^{11} -docosenoic acid (Langridge, 1977).

A crystal $0.12 \times 0.20 \times 0.36$ mm was used for data collection. The cell parameters were obtained by a least-squares analysis of the settings of 25 reflexions measured on a Philips PW 1100 four-circle diffractometer with Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å, graphite monochromated) and the ω – 2θ scan mode [scan width 1.1° (θ), scan speed 0.03° (θ) s^{-1}]. 4234 reflexions were collected in the range $3^\circ < \theta < 24^\circ$, of which 2514 unique reflexions had $I_{(rel)} > 2\sigma I_{(rel)}$ and were considered observed. The intensities of three standard reflexions measured every hour remained constant to within 1.1% of their mean values. Lorentz–polarization corrections were applied. No absorption correction was made. The I and P atoms were located in a Patterson map and a subsequent weighted difference Fourier synthesis yielded the positions of all but three of the nonhydrogen atoms. Several cycles of full-matrix least-squares refinement followed by a difference map gave the positions of the remaining nonhydrogen atoms and of 28 of the 38 H atoms. Owing to large thermal motion the bond lengths and angles in the aliphatic chain were very poor and we chose to overcome this problem by utilizing bond-length constraints in the final stages of refinement. Thus the C–C and C–O bonds were fixed at 1.54 and 1.42 Å respectively and the nonhydrogen atoms were treated anisotropically. The methyl H atoms were refined as rigid groups and the remaining H atoms constrained at 1.08 Å from their respective C atoms, their positions being dictated by the geometry of the molecule. The isotropic temperature factors of the H atoms, treated as three single parameters, refined to $U = 0.110$ Å² (aromatic H), $U = 0.157$ Å² (methylene H) and $U = 0.258$ Å² (methyl H). The refinement

converged to $R_w = \sum w^{1/2}|F_o - F_c| / \sum w^{1/2}|F_o| = 0.043$ and $R = 0.046$ with $w = 1/\sigma^2$. Final atomic coordinates for the nonhydrogen atoms are given in Table 1 and the relevant bond lengths and angles in Table 2.†

† Lists of structure factors, hydrogen atomic coordinates and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32918 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Fractional atomic coordinates ($\times 10^4$) of the heavy atoms

	<i>x</i>	<i>y</i>	<i>z</i>
I(1)	3743 (1)	1484 (0)	134 (0)
P(1)	3642 (2)	−193 (1)	7795 (1)
O(1)	12894 (23)	5974 (9)	9814 (8)
O(2)	12588 (10)	6417 (9)	8773 (5)
C(1)	4931 (7)	228 (4)	8443 (4)
C(2)	6208 (7)	590 (5)	8106 (3)
C(3)	7239 (8)	943 (6)	8707 (4)
C(4)	8463 (9)	1360 (6)	8368 (4)
C(5)	9182 (17)	1905 (10)	8960 (7)
C(6)	10179 (19)	2544 (10)	8692 (7)
C(7)	10168 (19)	3198 (11)	9278 (8)
C(8)	11259 (21)	3725 (10)	8936 (8)
C(9)	11397 (16)	4529 (9)	9345 (7)
C(10)	12440 (21)	4990 (10)	8911 (9)
C(11)	12604 (17)	5803 (9)	9302 (8)
C(12)	12966 (16)	7204 (8)	9008 (8)
Ring A			
C(21)	2959 (8)	553 (4)	7166 (4)
C(22)	1656 (9)	899 (5)	7237 (4)
C(23)	1147 (9)	1496 (6)	6762 (5)
C(24)	1907 (12)	1739 (5)	6205 (5)
C(25)	3219 (11)	1376 (5)	6124 (5)
C(26)	3727 (9)	801 (5)	6604 (4)
Ring B			
C(31)	2209 (8)	−559 (4)	8259 (4)
C(32)	2088 (9)	−401 (5)	8972 (4)
C(33)	912 (11)	−661 (6)	9289 (5)
C(34)	−140 (10)	−1080 (6)	8918 (6)
C(35)	−12 (10)	−1244 (6)	8201 (6)
C(36)	1159 (9)	−988 (5)	7873 (5)
Ring C			
C(41)	4395 (8)	−986 (4)	7321 (4)
C(42)	5347 (8)	−1507 (5)	7698 (4)
C(43)	5927 (9)	−2148 (5)	7355 (5)
C(44)	5570 (11)	−2279 (5)	6635 (5)
C(45)	4644 (10)	−1787 (6)	6241 (5)
C(46)	4038 (8)	−1134 (5)	6592 (4)

* Author to whom correspondence should be addressed.

Table 2. Selected bond lengths (Å) and bond angles (°)

C(1)—P(1)	1.788 (7)	C(21)—P(1)—C(1)	110.9 (3)
C(21)—P(1)	1.791 (7)	C(31)—P(1)—C(1)	108.7 (3)
C(31)—P(1)	1.785 (8)	C(31)—P(1)—C(21)	107.5 (4)
C(41)—P(1)	1.776 (8)	C(41)—P(1)—C(1)	110.1 (3)
C(11)—O(1)	1.01 (2)	C(41)—P(1)—C(21)	109.5 (3)
		C(41)—P(1)—C(31)	110.1 (4)

Discussion. The three phenyl rings and the aliphatic chain are tetrahedrally disposed about the phosphorus atom, the rings being orientated to reduce steric interaction (Fig. 1). The rings are all planar and the aliphatic

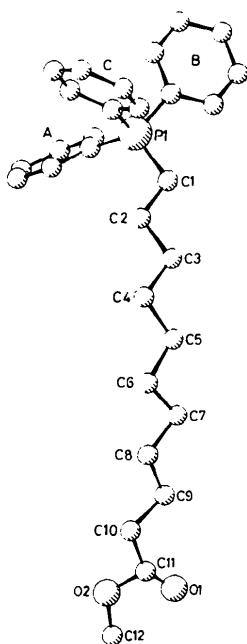
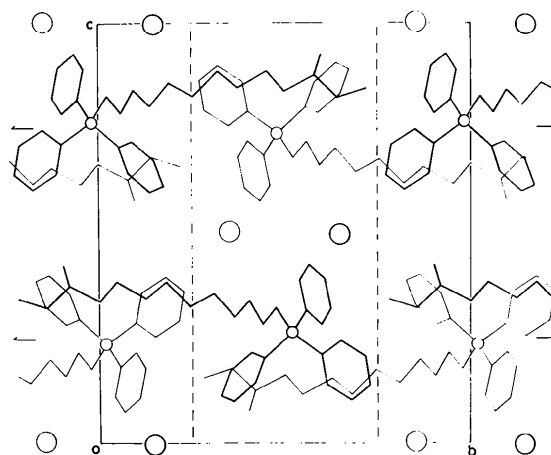


Fig. 1. A perspective view of the phosphonium cation.

Fig. 2. A projection of the structure viewed down a^* .

chain is totally extended in a wide open curve. The iodide anions lie in spaces between the phosphonium cations, the packing being relatively loose (Fig. 2). There are no intermolecular or interionic close contacts.

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Reference

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The Hydrogen Bonding of Artinite

BY M. AKAO AND S. IWAI

Research Laboratory for Engineering Materials, Tokyo Institute of Technology, Tokyo 152, Japan

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Abstract. $Mg_2CO_3(OH)_2(H_2O)_3$ (Utonai, Hokkaido, Japan), monoclinic, $C2/m$, $a = 16.560(5)$, $b = 3.153(1)$, $c = 6.231(3)$ Å, $\beta = 99.10(4)^\circ$, $Z = 2$, D_m (flotation) = 2.03, $D_x = 2.03$ g cm $^{-3}$. The H positions

have been determined from three-dimensional X-ray data. The water molecules are hydrogen-bonded to O atoms of the carbonate groups and to those of the hydroxyl groups.