bridge University IBM 370/165 computer and programs written by Dr G. M. Sheldrick; the figure was drawn with *PLUTO*, written by Dr W. D. S. Motherwell.

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Racemic 3-[Benzyl(phenyl)phosphinyl]-2-butenoic Acid

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Abstract. $C_{17}H_{17}O_3P$, $M_r = 300.3$, monoclinic, $P2_1/c$, Z = 4, a = 9.206 (3), b = 10.530 (4), c = 17.223 (5) Å, $\beta = 111.89$ (6)°, V = 1549.2 (Å)³, $D_x = 1.287$, $D_m = 1.282$ g cm⁻³ (by flotation in a mixture of benzene and bromobenzene), F(000) = 632. The structure was solved with *MULTAN* and refined by full-matrix leastsquares calculations to a final R of 0.066 ($R_w = 0.050$) for 2078 reflections measured on a Syntex diffractometer. Molecules with the same configuration form chains by means of strong hydrogen bonds of the type

$$P = 0 \cdots H - 0 - C - .$$

Introduction. The present study is a continuation of an investigation into the structure and hydrogen bonding in phosphine oxide derivatives containing a carboxylic group (Gałdecki & Główka, 1977*a,b*, 1978). It seemed interesting to determine the structure of a compound in which two groups taking part in the hydrogen bonding are separated by a rigid fragment (here the C=C double bond). Although Leiserowitz (1976, and references therein) discussed exhaustively the hydrogen bonding and geometry of α,β -unsaturated carboxylic acids in the crystal form, no analogy should be expected because of the different functional groups participating in the hydrogen bonding and their different positions in the molecule.

Prismatic crystals were obtained by the slow cooling of a saturated solution in a hot mixture of acetone and water. Intensities from a spherical crystal (about 0.3mm in diameter) were collected on a Syntex $P2_1$ fourcircle diffractometer with graphite-monochromated Cu $K\alpha$ radiation. Measurements were carried out in the θ - 2θ mode $(2\theta \le 114^{\circ})$. From 2078 recorded reflections, 273 had $F \le 2.0\sigma(F)$. The intensities were not corrected for absorption ($\mu = 12.6 \text{ cm}^{-1}$). The structure was solved by a multisolution technique (*MULTAN*, Germain, Main & Woolfson, 1971). The best solution was characterized by ABSFOM = 1.32, RESID = 29.32 and COMFOM = 3.00 for 356 $E \ge$ 1.4. The resulting *E* map revealed all the atoms but one (in the benzene ring) which was placed geometrically. The structure was then refined by full-matrix leastsquares calculations minimizing $\sum w(\Delta F)^2$. After four cycles of refinement with isotropic ($R_w = 0.121$) and a

Table 1. Atomic coordinates $(\times 10^4)$ of the nonhydrogen atoms

	x	У	Ζ
Р	8636 (2)	7707 (1)	2523(1)
O(1)	7976 (4)	6398 (4)	2336 (3)
O(2)	4525 (5)	10213 (4)	2724 (3)
O(3)	3066 (5)	8624 (5)	2029 (4)
C(1)	214 (7)	7813 (6)	3523 (4)
C(2)	45 (8)	7100 (8)	4177 (5)
C(3)	1250 (10)	7165 (9)	4994 (5)
C(4)	2568 (10)	7945 (9)	5132 (5)
C(5)	2711 (9)	8625 (8)	4464 (6)
C(6)	1537 (8)	8573 (7)	3660 (5)
C(7)	7207 (7)	8883 (6)	2563 (4)
C(8)	5759 (7)	8423 (6)	2345 (4)
C(9)	4319 (7)	9116 (6)	2339 (4)
C(10)	9418 (7)	8284 (6)	1756 (4)
C(11)	8170 (7)	8191 (7)	881 (4)
C(12)	7396 (8)	9299 (7)	484 (5)
C(13)	6358 (10)	9211 (9)	-352 (5)
C(14)	5930 (9)	8052 (10)	-752 (5)
C(15)	6698 (10)	6939 (9)	-365 (5)
C(16)	7820 (8)	7012 (7)	457 (5)
C(17)	7812 (8)	10219(6)	2848 (5)

P-

further three cycles with anisotropic temperature factors, R was 0.081 ($R_w = 0.073$). At this stage of the calculation, a difference synthesis revealed the positions of all the H atoms which were included together with individual isotropic temperature factors in the final two cycles of refinement. Non-hydrogen atoms were held fixed. The final values of R and R_{w} were 0.066 and 0.050 respectively. The weights were $w^{-1} = \sigma(F)$. Neutral-atom scattering factors (Doyle & Turner, 1968) were employed. All the calculations were made using the XRAY 70 system (Stewart, Kundell & Baldwin, 1970) on an IBM 370/145 computer.*

Discussion. The heavy-atom coordinates are listed in Table 1, and H atom positional parameters and isotropic temperature factors are presented in Table 2.

Table 2. Hydrogen atom positional $(\times 10^3)$ and thermal $(Å^2 \times 10^3)$ parameters, and H-X bond lengths (X = C, O

				Bonded		
	x	У	z	$U_{\rm iso}$	to	$H-X(\dot{A})$
H(1)	341 (7)	1059 (6)	260 (3)	212 (26)	$\begin{cases} O(2) \\ O(1) \end{cases}$	1.04(3)
H(2)	902 (8)	648 (6)	410 (4)	212 (29)	C(2)	1.38(4) 1.12(3)
H(3)	1112 (8)	659 (6)	542 (4)	244 (32)	C(3)	0.99 (4)
H(4)	1353 (7)	795 (5)	570 (3)	155 (23)	C(4)	1.04 (5)
H(5)	1363 (6)	929 (5)	454 (3)	135 (21)	C(5)	1.07(1)
H(6)	1169 (6)	912 (5)	316 (3)	113 (18)	C(6)	1.09 (6)
H(8)	547 (5)	739 (4)	206 (2)	61 (14)	C(8)	1.18 (5)
H(101)	977 (5)	930 (5)	185 (3)	108 (18)	C(10)	1.12 (4)
H(102)	1035 (5)	769 (4)	179 (2)	77 (15)	C(10)	1.04 (2)
H(12)	764 (5)	1018 (5)	82 (3)	108 (19)	C(12)	1.07(1)
H(13)	571 (7)	1012 (6)	-60(3)	165 (24)	C(13)	1.09 (2)
H(14)	511 (5)	800 (4)	-138(3)	79 (16)	C(14)	1.06 (4)
H(15)	647 (8)	597 (7)	-72 (4)	249 (33)	C(15)	1.17 (7)
H(16)	834 (6)	616 (5)	74 (3)	158 (24)	C(16)	1.12 (4)
H(171)	871 (7)	36 (5)	273 (3)	189 (23)	C(17)	0.93 (7)
H(172)	705 (7)	82 (6)	275 (4)	150 (25)	C(17)	0.91 (8)
H(173)	831 (8)	16 (6)	362 (4)	198 (30)	C(17)	1.23(9)

Table 3. Bond lengths (Å)

P-O(1)	1.493 (1)	C(7)–C(17)	1.525 (3)
P-C(1)	1.796 (2)	C(8) - C(9)	1.510(1)
P-C(7)	1.827 (2)	C(9)–O(2)	1.310(1)
P - C(10)	1.827 (1)	C(9)-O(3)	1.194 (1)
C(1) - C(2)	1.410(1)	C(10)-C(11)	1.520(1)
C(2) - C(3)	1.431 (1)	C(11)–C(12)	1.404 (1)
C(3) - C(4)	1.415 (1)	C(12)–C(13)	1.434 (2)
C(4) - C(5)	1.402 (1)	C(13)C(14)	1.379 (5)
C(5) - C(6)	1.405 (1)	C(14)C(15)	1.402 (3)
C(6) - C(1)	1.402 (1)	C(15)–C(16)	1.409 (2)
C(7) - C(8)	1.333 (1)	C(16)–C(11)	1.425 (3)

Bond lengths and angles are in Tables 3 and 4. The atom numbering is shown in Fig. 1.

As expected, a considerable part of the molecule is almost flat as a result of the conjugated system of double bonds.



The shortening of the C(8)-C(9) bond is noticeable (1.510 Å). The angle between the plane of the carboxyl group and the plane of the double bond C(7)=C(8) is about 13° (Table 5). The planar fragments of the molecules having the same configuration, linked into long chains along the screw axis by means of hydrogen bonding, are nearly parallel to each other (Fig. 2). Phenyl and benzyl substituents fill the space between the pseudolayers formed from the chains. It is interesting that the antiplanar C=C-C=O conformation is observed; this was found in none of the

Table 4. Bond angles (°)

P-C(7)-C(8)	113.64 (20)	C(7)-C(8)-C(9)	127.50 (14)
P-C(7)-C(17)	117.09 (18)	C(8) - C(7) - C(17)	129.26 (19)
P-C(10)-C(11)	109.85 (2)	C(8)-C(9)-O(2)	117-48 (5)
P - C(1) - C(2)	116-28 (2)	C(8)-C(9)-O(3)	119-51 (9)
P-C(1)-C(6)	122-52 (5)	O(2)-C(9)-O(3)	122-89 (8)
C(1) - P - O(1)	112.48 (2)	C(10)-C(11)-C(12)	119.18 (1)
C(1) - P - C(7)	104-96 (5)	C(10)–C(11)–C(16)	120.71 (7)
C(1) - P - C(10)	106.06 (3)	C(11)-C(12)-C(13)	118-93 (18)
O(1) - P - C(7)	113-17 (8)	C(12)-C(13)-C(14)	120.01 (3)
O(1) - P - C(10)	112.93 (1)	C(13)-C(14)-C(15)	121.68 (28)
C(7) - P - C(10)	106-61 (7)	C(14)-C(15)-C(16)	118.88 (32)
C(1)-C(2)-C(3)	119-11 (4)	C(15)-C(16)-C(11)	120-42 (12)
C(2)-C(3)-C(4)	119-46 (6)	C(16)-C(11)-C(12)	120.08 (8)
C(3) - C(4) - C(5)	119.88 (4)		
C(4) - C(5) - C(6)	121.07 (3)	C(9) - O(2) - H(1)	106-54 (39)
C(5)-C(6)-C(1)	119-24 (5)	$P-O(1)\cdots H(1)$	139.50 (44)
C(6)-C(1)-C(2)	$121 \cdot 19(5)$		



Fig. 1. A view of the molecule and the numbering of the atoms (ORTEP, Johnson, 1965).

^{*} Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33585 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 5. Least-squares planes

(a) Coefficients in AX + BY + CZ = D, referred to the crystallographic axes

A	В	С	D		
Plane (I): C(1)(6)					
-5.4781	8.1277	7.3993	3.3573		
Plane (II): C(11)-(16)					
-8.4425	-1.7010	11.7144	-7.2596		
Plane (III): C(9), O(2), O(3)					
-1.7832	-4-9288	15.0220	-1.7504		
Plane (IV): C(7), C(8), C(9)					
-1-4942	-2.9696	16.1538	0.4260		
Plane (V): C(7), C(8), C(17)					
-1.5827	-2.7563	16.2813	0.5846		
Plane (VI): P, C(7), C(8)					
-1.6546	-2.5742	16.3819	0.7201		
(b) Deviations of the atoms from the planes $(Å)$					

	(I)	(II)	(III)	(IV)	(V)	(VI)
Р	0.0431			0.0703	0.0320	
C(1)	0.0042			0 0 0		
C(2)	0.0013					
C(3)	-0.0090					
C(4)	0.0120					
C(5)	-0.0072					
C(6)	-0.0012					
C(7)			-0.0632			
C(8)			0.0933			
C(9)					0.0276	0-0505
C(10)		-0.0432				
C(11)		0.0004				
C(12)		0.0010				
C(13)		-0.0029				
C(14)		0.0037				
C(15)		-0.0025				
C(16)		0.0003				
C(17)				-0.0267		0.0230
H(1)			-0.1734			
(c) Angles between planes (°)						
	(III)/(IV)	11-8:	5	(III)/(VI)	13.89	
	(III)/(V)	12.93	3			

previously examined $R-CH_2-CH_2-COOH$ monocarboxylic acids and was only rarely present in α,β unsaturated ones (Leiserowitz, 1976). A similar arrangement has only been found in *trans*-cinnamic acid (Ladell, McDonald & Schmidt, 1956) and fumaric acid (Brown, 1966; Bednowitz & Post, 1966). A synplanar conformation is more usually found.



Fig. 2. The packing of the molecules in the racemic crystal.

As in the organic phosphorus compounds of the type $R_3P=O$ previously studied, a deformation of the P coordination tetrahedron (comprising an increase in the O=P-C and a simultaneous decrease in the C-P-C valency angles) is found. In the crystal studied the mean values of these angles are about 106 and 113°.

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