An intramolecular H bridge between N and O(4) [N···O(4) = 2·776 (3), H(5)···O(4) = 2·09 (4) Å] closes a planar six-membered ring comprising N, C(7), C(10), C(11), O(4) and H(5). The ethyl group has a staggered conformation and lies close to the plane of the dihydrofuran [torsion angles: O(4)–C(11)–O(3)–C(12) = −0·5 (4), C(10)–C(11)–O(3)–C(12) = −179·9 (2), C(11)–O(3)–C(12)–C(13) = 175·0 (2)°].

The phenyl ring itself is planar. The Cl and N atoms, however, show significant deviations of 0·074 (4) and 0·105 (4) Å, respectively, from the mean plane through atoms C(1) to C(6). This seems to be typical for disubstituted benzenes and has been found, for example, in p-chloroaniline (Trotter, Whitlow & Zobel, 1966; Palm, 1966), p-nitrophenol (Coppens & Schmidt, 1965), p-dichlorobenzene (Housty & Clastre, 1957), o-chlorobenzoic acid (Ferguson & Sim, 1961), o-bromobenzoic acid (Ferguson & Sim, 1962) and 2,5-dichloroaniline (Sakurai, Sundaralingam & Jeffrey, 1963).

Repulsion between O(1) and H(3) causes a rotation of the phenyl ring through 23·9 (2)° about the N–C(4) bond away from the plane of the heterocyclic ring [torsion angles: C(7)–N–C(4)–C(5) = −21·3 (4), C(7)–N–C(4)–C(3) = 162·5 (3), H(5)–N–C(4)–C(3) = −23·2 (2·6) and H(5)–N–C(4)–C(5) = 153·0 (2·6)°].

This relatively small torsion, in conjunction with the position of the ester group in the molecular plane, allows a close packing of rather flat molecules parallel to (10$\bar{4}$), largely determined by van der Waals forces.

There are six molecules along the c translation, giving 3·46 Å per molecule, in agreement with the 'thickness' of benzene (3·2 Å) and the space required for Cl (3·5 Å). Each molecule has 12 nearest neighbours; there are no intermolecular contacts closer than expected from the van der Waals radii.

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Methyl [α-(2-Furoyl)benzyl]phenylphosphinate, C19H17O4P

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Abstract. $M_r = 340·31$, orthorhombic, Pbca, $a = 18·398$ (9), $b = 16·700$ (8), $c = 11·034$ (6) Å, $V = 3390$ (2) Å^3, $Z = 8$, $D_x = 1·33$ Mg m^{-3}, Cu Kα, $\lambda = 1·5418$ Å, $\mu = 1·594$ mm^{-1}, $F(000) = 1424$, room temperature, final $R = 0·066$ for 1008 reflections ($F \geq 3\sigma$, $\sin\theta/\lambda \leq 0·48$ Å^{-1}) and 268 refined parameters. The molecule adopts a staggered conformation. The planes of the two phenyl rings are rotated through 21·7 (8)° with respect to each other. The dihedral angles between the furan, which is planar, and the phenyl rings are 73·2 (8) and 74·7 (8)°, respectively.

Introduction. Photolysis of α-carbonyl-α'-phosphoryl-substituted diazo compounds yields the corresponding carbenes, which normally re-arrange to both ketenes and phosphines (Regitz, 1975). In the case of the 2-furoyl-substituted diazo compounds, however, only the phosphines were observed. These may be stabilized

with methanol to yield two diastereomeric phosphinates, corresponding to the R,S and R,R forms. If the configuration of one of the two reaction products is known, the configuration of other differently substituted phosphinates can then be assigned from characteristic signals of their ^{1}H NMR spectra.

Table 1. *Fractional atomic coordinates* $(\times 10^4, \times 10^3$ *for* H) *and isotropic thermal parameters (for* H *fixed at the value of the parent atom), with e.s.d.'s in units of the least significant digit in parentheses*

$$B = \tfrac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i\mathbf{a}_j.$$

	x	y	z	$B(\text{Å}^2)$
P	8912 (1)	6384 (1)	5410 (2)	3·7 (1)
O(1)	9005 (3)	5926 (4)	6669 (6)	4·7 (3)
O(2)	9086 (3)	5903 (3)	4341 (6)	4·6 (3)
O(3)	7985 (3)	7401 (4)	3695 (5)	4·9 (3)
O(4)	6971 (3)	8485 (3)	4403 (6)	4·7 (3)
C(1)	9452 (5)	7282 (5)	5554 (8)	3·8 (5)
C(2)	9973 (5)	7445 (6)	4682 (9)	5·5 (7)
C(3)	10406 (6)	8113 (6)	4800 (9)	5·9 (6)
C(4)	10315 (6)	8628 (6)	5763 (9)	5·6 (5)
C(5)	9804 (5)	8478 (6)	6633 (9)	4·8 (5)
C(6)	9362 (5)	7808 (5)	6539 (8)	4·4 (5)
C(7)	9719 (5)	5620 (6)	6958 (11)	6·5 (7)
C(8)	7966 (4)	6686 (5)	5608 (8)	3·6 (4)
C(9)	7449 (5)	5975 (5)	5433 (8)	3·3 (4)
C(10)	7095 (5)	5671 (5)	6431 (8)	4·1 (5)
C(11)	6645 (5)	5027 (6)	6317 (9)	5·3 (5)
C(12)	6543 (5)	4674 (6)	5173 (10)	5·2 (6)
C(13)	6898 (5)	4980 (5)	4185 (8)	3·8 (5)
C(14)	7351 (5)	5629 (5)	4300 (8)	3·9 (5)
C(15)	7765 (4)	7376 (5)	4747 (7)	3·4 (4)
C(16)	7246 (5)	7949 (5)	5230 (8)	3·9 (5)
C(17)	6995 (6)	8115 (6)	6328 (8)	4·8 (6)
C(18)	6541 (6)	8806 (6)	6204 (10)	5·6 (6)
C(19)	6534 (5)	9000 (5)	5039 (9)	4·8 (6)
H(1)	994 (6)	693 (6)	420 (9)	5·5
H(2)	1079 (6)	818 (6)	400 (9)	5·9
H(3)	1069 (6)	908 (6)	610 (9)	5·6
H(4)	971 (5)	891 (6)	724 (9)	4·8
H(5)	890 (6)	760 (6)	716 (9)	4·4
H(6)	978 (6)	540 (6)	771 (10)	6·5
H(7)	997 (6)	519 (6)	621 (9)	6·5
H(8)	1015 (6)	610 (6)	712 (10)	6·5
H(9)	793 (5)	699 (5)	639 (8)	3·6
H(10)	720 (5)	600 (6)	718 (9)	4·1
H(11)	645 (6)	469 (6)	694 (9)	5·3
H(12)	615 (6)	414 (6)	502 (9)	5·2
H(13)	677 (5)	467 (6)	326 (9)	3·8
H(14)	758 (6)	590 (6)	364 (9)	3·9
H(15)	709 (5)	779 (5)	707 (9)	4·8
H(16)	626 (6)	895 (6)	696 (10)	5·6
H(17)	651 (5)	964 (6)	471 (9)	4·8

Table 2. *Intramolecular bond distances* (Å) *and angles* (°) *not involving hydrogen, with e.s.d.'s in parentheses* (*Busing, Martin & Levy,* 1964)

P—O(1)	1·595 (7)	P—O(2)	1·462 (7)
P—C(1)	1·805 (9)	P—C(8)	1·825 (9)
O(1)—C(7)	1·445 (12)	O(3)—C(15)	1·230 (10)
O(4)—C(16)	1·374 (11)	O(4)—C(19)	1·371 (11)
C(8)—C(9)	1·534 (12)	C(8)—C(15)	1·539 (12)
C(15)—C(16)	1·452 (12)	C(16)—C(17)	1·326 (13)
C(17)—C(18)	1·431 (14)	C(18)—C(19)	1·326 (15)

Range for aromatic C—C: 1·366 (15)–1·407 (13) Å, mean 1·381 (4) Å

O(2)—P—O(1)	114·6 (4)	C(1)—P—O(1)	105·3 (4)
C(8)—P—O(1)	97·5 (4)	C(7)—O(1)—P	117·3 (6)
C(1)—P—O(2)	114·0 (4)	C(8)—P—O(2)	117·2 (4)
C(8)—P—C(1)	106·6 (4)	C(2)—C(1)—P	118·9 (7)
C(6)—C(1)—P	121·4 (7)	C(9)—C(8)—P	111·3 (6)
C(15)—C(8)—P	111·3 (6)	C(8)—C(15)—O(3)	121·9 (8)
C(16)—C(15)—O(3)	122·7 (8)	C(19)—O(4)—C(16)	106·6 (7)
C(15)—C(16)—O(4)	115·3 (7)	C(17)—C(16)—O(4)	110·0 (8)
C(18)—C(19)—O(4)	109·6 (8)	C(15)—C(8)—C(9)	110·7 (7)
C(16)—C(15)—C(8)	115·2 (7)	C(17)—C(16)—C(15)	134·5 (9)
C(18)—C(17)—C(16)	106·5 (9)	C(19)—C(18)—C(17)	107·3 (9)

Range for aromatic C—C—C: 118·6 (8)–120·7 (9)°, mean 119·9 (3)°

Experimental. Sample crystal $(0.28 \times 0.12 \times 0.40$ mm) mounted on paper-tape-controlled four-circle Siemens AED diffractometer, [001] along spindle axis; Ni-filtered Cu radiation; cell constants refined from 12 centred reflections $(43.4 \le 2\theta \le 88.6°)$; ω–2θ scan at $2.5°$ min^{-1}, scan width $1° + \alpha_1/\alpha_2$ splitting, same time spent on peak and background; 1514 unique data measured with $(\sin\theta/\lambda)_{max} = 0.48$ Å^{-1} (1/8 sphere, $h \le 17$, $k \le 15$, $l \le 10$); two test reflections (10,0,0 and 080) showed only random fluctuations ($<3\%$); intensities corrected for background and Lp factor, absorption ignored. Direct methods (Main, Woolfson, Lessinger, Germain & Declercq, 1974) for structure solution, H atoms located in difference Fourier synthesis; refinement by block-diagonal least squares (Bartlett, 1972), minimizing $\sum w\Delta^2 = \sum w(|F_o| - K|F_c|)^2$; unit weights, producing fairly constant $\langle w\Delta^2\rangle$ over ranges in $|F_o|$. One scale factor, atomic coordinates and anisotropic thermal parameters for non-H's adjusted, temperature factors of hydrogens kept fixed at isotropic value of parent atom; convergence at $R = 0.066$, $wR = 0.069$, $S = 3.099$ for 1008 observations $(F_o \ge 3\sigma)$ and 268 variables; largest shift 0.2σ. Two reflections (002, 412) with $w\Delta > 10$ discarded; no evidence of secondary extinction. Scattering factors for neutral atoms according to Onken & Fischer (1968). Final $\Delta\rho$ ranging from -0.17 to 0.16 e Å^{-3} without any significant features.

Discussion. Table 1 lists coordinates and (equivalent) isotropic thermal parameters, Table 2 gives bond lengths and angles; the molecular structure and labelling scheme are shown in Fig. 1.*

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* Lists of structure factors, anisotropic thermal parameters, bond data involving H atoms, least-squares planes and torsion angles around the P—C bond have been deposited with the British Library Lending Division as Supplementary Publication No.SUP 39765 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
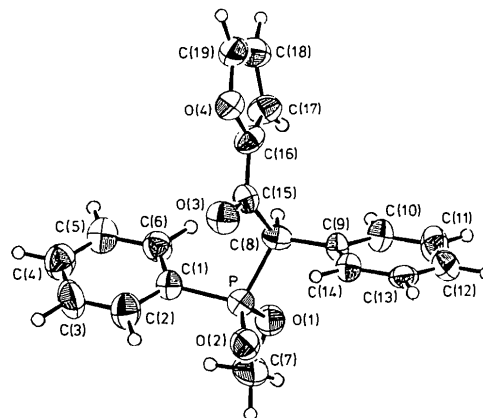

Fig. 1. Perspective view of the molecule and atomic numbering. The thermal ellipsoids of the non-H's are for 50% probability (Johnson, 1965).

The molecule has the *R,S* configuration and adopts a staggered conformation. The phenyl rings and the furoyl group are planar, the maximum deviations from the best planes being 0·005 (9), 0·001 (8) and 0·007 (10) Å respectively. The aromatic C—C lengths average 1·381 (4) Å, the C—H lengths 1·06 (2) Å (excluding methyl H's). The molecular structure displays no unusual features.

The thermal motion of the molecule appears to be governed by a libration about the P—C bond and wagging motions of the furan and phenyl groups. Atoms P and C(8) vibrate almost isotropically, the r.m.s. vibration components along the crystallographic axes being 0·214 (7), 0·209 (7), 0·226 (7) Å for P and 0·204 (27), 0·229 (25), 0·209 (26) Å for C(8).

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Structure of 3-Benzyl-5-(methoxycarbonylmethylthiomethyl)hydantoin,* $C_{14}H_{16}N_2O_4S$†

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Abstract. $M_r = 308·4$, monoclinic, $P2_1$, $a = 11·158$ (2), $b = 6·010$ (1), $c = 11·428$ (3) Å, $\beta = 98·80$ (2)°, $V = 757·3$ Å^3, $Z = 2$, $D_m = 1·330$, $D_x = 1·352$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0·7107$ Å, $\mu = 0·23$ mm^{-1}, $F(000) = 324$, $T = 293$ K. $R = 0·037$ for 1039 observed reflections. The hydantoin ring is planar and the dihedral angle it makes with the aromatic ring is 89·3 (7)°. There is a hydrogen bond N—H···O, 2·89 (1) Å, linking the molecules along the y axis, *via* N(1) and the C(4) carbonyl oxygen atom.

Introduction. As a part of the project on the synthesis of (+)-biotin from L(+)-cysteine, the optically active title compound has been prepared (Ravindranathan, Hiremath & Reddy, 1984).

Experimental. Crystal 0·40 × 0·12 × 0·35 mm, D_m by flotation in aqueous KBr solution, Nonius CAD-4F-11M diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan mode, scan speed 1° min^{-1}, $\theta < 23·5°$, h 0 to 12, k 0 to 6, l −12 to 12. 1369 reflections collected, 1039 judged significant ($|F_o| > 3\sigma|F_o|$), lattice parameters from 19 reflections

($16 < 2\theta < 38°$), three standard reflections (400, 004 and 31$\bar{3}$) every 2000 s, 4% variation in intensity. No correction for absorption. Structure solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least-squares refinement of scale factor, positional and anisotropic thermal parameters (isotropic thermal parameters for hydrogen atoms, initial hydrogen positions calculated), final $R = 0·037$, $wR = 0·035$, $S = 0·72$, $\sum w(|F_o| - |F_c|)^2$ minimized, $w = (2·5 + 1·0|F_o| + 0·04|F_o|^2)^{-1}$. $(\Delta/\sigma)_{max} = 0·3$. Final $\Delta\rho$ excursions < |0·2| e Å^{-3}. No correction for secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Programs *LALS* (Gantzel, Sparks & Trueblood, 1961) used for refinement, *NRC*-12 and *NRC*-22 (Ahmed, Hall, Pippy & Huber, 1973) for bond lengths and angles and torsion angles.

Discussion. The atomic coordinates with their e.s.d.'s and equivalent isotropic temperature factors are given in Table 1.§ Bond lengths and bond angles involving the

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