from planarity for the other ring atoms is shown by N(2) and is 0·035 (4) Å. This distortion is ascribed to steric repulsions between the phosphazene ring atoms and the methyl group attached to the other ring.

The mean P—N bond distance at P(1) of 1·608 (3) Å is longer than those adjacent to the $PCl_2$ units. Of the latter, the mean P—N bond distance closest to the P—P bridge is 1·562 (3) Å while the other is 1·572 (3) Å. This alternation in longer and shorter P—N bonds has been detected for other geminally substituted cyclo-triphosphazenes and has been attributed to the influence by the less electronegative substituents at P(1) on the $\pi$ bonding in the ring (Mani, Ahmed & Barnes, 1965; Craig & Paddock, 1962).

The unequal bond lengths within the six-membered ring lead to corresponding distortions of the endocyclic bond angles from 120°. The largest deviation is at P(1) where the N(1)—P(1)—N(3) bond angle is narrowed to 115·8 (1)°. Both steric and electronic arguments have been used to explain this effect (Allcock, Connolly & Whittle, 1983; Ritchie, Harris & Allcock, 1980).

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# Structure of (*RS–SR*)-Ethyl 2,5-Dioxo-4-phenyl-3-pyrrolidinecarboxylate, $C_{13}H_{13}NO_4$

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**Abstract.** $M_r = 247·25$, monoclinic, $P2_1/n$, $a = 15·286$ (3), $b = 5·377$ (2), $c = 16·218$ (4) Å, $\beta = 109·72$ (2)°, $V = 1254·8$ Å$^3$, $Z = 4$, $D_x = 1·309$ Mg m$^{-3}$, $\lambda(Mo\ K\alpha_1) = 0·70930$ Å, $\mu = 0·091$ mm$^{-1}$, $F(000) = 520$, $T = 291$ K, $R = 0·046$ for 1130 observed reflexions. The analysis shows the compound to be a *trans*-substituted succinimide, hydrogen-bonded into one-dimensional chains [N(H)···O = 2·870 (4) Å] running in the **b** direction. The five-membered ring is in an envelope conformation with the phenyl-substituted carbon atom lying 0·224 (3) Å out of the least-squares plane through the other four atoms.

**Introduction.** We recently published a preliminary report on work performed on the molybdenum carbonyl induced reaction of azirines with carbanions derived from $\beta$-dicarbonyl compounds (Alper, Mahatantila, Einstein & Willis, 1984). The following reaction was found to take place.
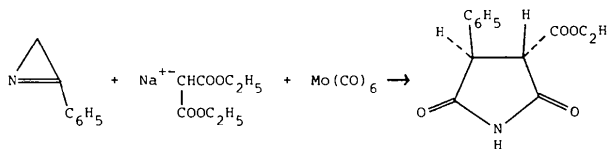


An X-ray structure analysis performed to establish the identity of the product is reported here.

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**Experimental.** White needle, $0.22 \times 0.46 \times 0.12$ mm, Nonius CAD-4F diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, lattice parameters from setting angles of 25 reflexions with $19 < 2\theta < 28°$, absorption ignored, $\theta$–$2\theta$ scan, $h$ $0 \rightarrow 16$, $k$ $0 \rightarrow 5$, $l$ $-17 \rightarrow 16$ with $0 < 2\theta < 45°$, variations in intensities of two standards <2%, 1632 independent reflexions, 1130 with $I > 2.3\sigma(I)$, Lp correction applied, *MULTAN* full-matrix refinement on $F$ with anisotropic temperature factors except H atoms (from $\Delta F$ synthesis) isotropic, final $R = 0.046$, $wR = 0.055$, $S = 1.84$, $w = \{[\sigma(F)]^2 + 0.0004F^2\}^{-1}$, all $\Delta/\sigma < 0.1$ in final cycle, all features in final $\Delta F$ synthesis between $\pm 0.19$ (3) e Å$^{-3}$, VAX 11/750 computer (Larson & Gabe, 1978); scattering factors from *International Tables for X-ray Crystallography* (1974).

**Discussion.** An *ORTEP* diagram (Johnson, 1970) of the molecule displaying the atom labelling is shown in Fig. 1. The final atomic coordinates of the non-hydrogen atoms are given in Table 1 and selected bond lengths and angles in Table 2.*

The structure analysis reveals the product to be succinimide, *trans*-substituted by an ethyl ester and a phenyl group at the 3 and 4 positions respectively.

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* A stereoscopic view of the unit-cell contents, hydrogen-atom coordinates, anisotropic temperature factors, additional bond lengths and angles, intermolecular contacts, torsion angles, least-squares planes and a list of observed and calculated structure factors have been deposited with the British Library Lending Division as Supplementary Publiction No. SUP 39864 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
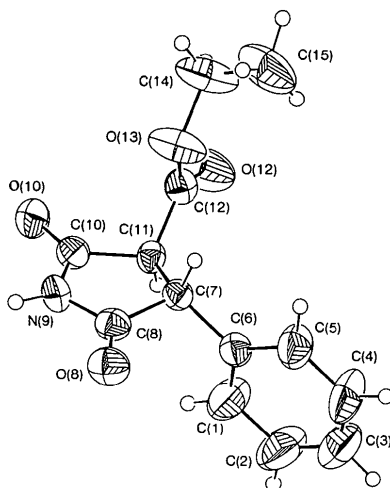


Fig. 1. *ORTEP* diagram of the molecule showing atomic labelling. Thermal ellipsoids enclose 50% probabilities; hydrogen atoms are shown as arbitrarily small circles.

Table 1. *Fractional atomic coordinates and* $B_{eq}$ *of non-hydrogen atoms*

E.s.d.'s refer to the last digit printed.

| | $x$ | $y$ | $z$ | $B_{eq}{}^*$(Å$^2$) |
|---|---|---|---|---|
| C(1) | 0.0428 (4) | 0.2148 (10) | 0.1150 (3) | 6.3 (3) |
| C(2) | 0.0968 (4) | 0.2206 (12) | 0.0617 (3) | 7.6 (3) |
| C(3) | 0.1712 (4) | 0.0729 (12) | 0.0784 (4) | 6.7 (3) |
| C(4) | 0.1919 (4) | −0.0844 (12) | 0.1470 (4) | 7.3 (3) |
| C(5) | 0.1389 (3) | −0.0926 (9) | 0.2008 (3) | 5.4 (2) |
| C(6) | 0.0630 (2) | 0.0578 (7) | 0.1849 (2) | 3.5 (2) |
| C(7) | 0.0040 (2) | 0.0499 (7) | 0.2432 (2) | 3.0 (2) |
| C(8) | −0.0917 (3) | −0.0592 (8) | 0.1990 (2) | 3.5 (2) |
| O(8) | −0.1121 (2) | −0.2383 (5) | 0.1512 (2) | 4.7 (1) |
| N(9) | −0.1545 (2) | 0.0806 (7) | 0.2245 (2) | 3.9 (2) |
| C(10) | −0.1168 (3) | 0.2850 (7) | 0.2724 (2) | 3.5 (2) |
| O(10) | −0.1571 (2) | 0.4331 (6) | 0.3039 (2) | 4.8 (1) |
| C(11) | −0.0156 (2) | 0.3003 (7) | 0.2787 (2) | 3.0 (2) |
| C(12) | 0.0453 (3) | 0.3702 (8) | 0.3692 (2) | 3.5 (2) |
| O(12) | 0.0854 (2) | 0.5612 (6) | 0.3886 (2) | 6.9 (2) |
| O(13) | 0.0490 (2) | 0.1910 (5) | 0.4249 (2) | 6.2 (2) |
| C(14) | 0.1074 (4) | 0.2229 (11) | 0.5167 (3) | 7.1 (3) |
| C(15) | 0.1834 (4) | 0.0539 (12) | 0.5387 (3) | 8.4 (3) |

* $B_{eq}$ is the arithmetic mean of the principal axes of the thermal ellipsoid.

Table 2. *Selected interatomic distances* (Å) *and angles* (°)

| | | | | |
|---|---|---|---|---|
| C(1)–C(2) | 1.381 (7) | | C(8)–N(9) | 1.388 (5) |
| C(2)–C(3) | 1.338 (9) | | N(9)–C(10) | 1.357 (5) |
| C(3)–C(4) | 1.348 (9) | | C(10)–O(10) | 1.219 (5) |
| C(4)–C(5) | 1.377 (7) | | C(10)–C(11) | 1.519 (5) |
| C(5)–C(6) | 1.365 (6) | | C(11)–C(12) | 1.497 (5) |
| C(6)–C(1) | 1.364 (6) | | C(12)–O(12) | 1.183 (5) |
| C(6)–C(7) | 1.511 (5) | | C(12)–O(13) | 1.308 (5) |
| C(7)–C(8) | 1.513 (5) | | O(13)–C(14) | 1.465 (5) |
| C(7)–C(11) | 1.533 (5) | | C(14)–C(15) | 1.422 (9) |
| C(8)–O(8) | 1.209 (5) | | | |

| | | | | |
|---|---|---|---|---|
| C(6)–C(1) C(2) | 121.3 (5) | | O(8)–C(8)–N(9) | 124.6 (4) |
| C(1)–C(2)–C(3) | 120.4 (5) | | C(8)–N(9)–C(10) | 114.0 (3) |
| C(2)–C(3)–C(4) | 119.2 (5) | | N(9)–C(10)–O(10) | 126.3 (3) |
| C(3)–C(4)–C(5) | 121.2 (5) | | N(9)–C(10)–C(11) | 108.0 (3) |
| C(4)–C(5)–C(6) | 120.3 (5) | | O(10)–C(10)–C(11) | 125.7 (4) |
| C(5)–C(6)–C(1) | 117.6 (4) | | C(7)–C(11)–C(10) | 104.6 (3) |
| C(5)–C(6)–C(7) | 120.9 (4) | | C(7)–C(11)–C(12) | 117.0 (3) |
| C(1)–C(6)–C(7) | 121.5 (4) | | C(10)–C(11)–C(12) | 111.2 (3) |
| C(6)–C(7)–C(8) | 113.6 (3) | | C(11)–C(12)–O(12) | 125.0 (4) |
| C(6)–C(7)–C(11) | 116.3 (3) | | C(11)–C(12)–O(13) | 111.1 (3) |
| C(8)–C(7)–C(11) | 103.8 (3) | | O(12)–C(12)–O(13) | 123.9 (4) |
| C(7)–C(8)–O(8) | 127.7 (4) | | C(12)–O(13)–C(14) | 119.0 (4) |
| C(7)–C(8)–N(9) | 107.7 (3) | | O(13)–C(14)–C(15) | 110.2 (4) |

Bond lengths and angles within the five-membered ring closely parallel those found in succinimide (Mason, 1961) and the carbon-substituted derivatives 3-phenyl-succinimide (Argay & Kálmán, 1973) and *trans*- and *cis*-3-(1-methoxyethyl)-4-methylsuccinimide (Sheldrick, 1981a,b). They are well within the range expected for $sp^3$ and $sp^2$ carbon, nitrogen and oxygen atoms. There is no obvious chemical reason for the small asymmetry in the C—N distances but it may result from the fact that this nitrogen atom is involved in an intermolecular hydrogen bond.

Several of the peripheral atoms of the substituent groups show somewhat unreasonable anisotropic thermal parameters (see Fig. 1) which could be interpreted as indicating some degree of disorder. Interatomic

distances involving these atoms are consequently less reliable.

The succinimides mentioned above were found to be linked into dimers by pairs of hydrogen bonds between the imide hydrogen atoms and carbonyl oxygen of adjacent molecules with $N(H)\cdots O$ distances of about 2·88 Å. In the present compound there is a corresponding contact distance of 2·870 (4) Å for $N(9)\cdots O(10)$, but this time the molecules are linked into a one-dimensional chain running in the **b** direction.

Examination of least-squares planes through the atoms of the five-membered ring shows that it has an envelope conformation (Bucourt, 1974) with C(7) out of plane (flap angle 13·7°). Deviations (in Å, e.s.d.'s = 0·003 Å) from the least-squares plane of atoms C(8)–C(11) are: C(8), −0·009; N(9), 0·015; C(10), −0·014; C(11), 0·008; other atoms: C(7), −0·224; O(8), 0·101; O(10), −0·032.

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# Benzoate de Décahydro-1,2,3,4,5,6,6a,7,8,11 Triméthyl-4,6a,9 Méthano-8,11a 11a*H*-Cyclohepta[*a*]naphtyl-4 Méthyle-(4α,6aβ,8β,11aα), C27H34O2

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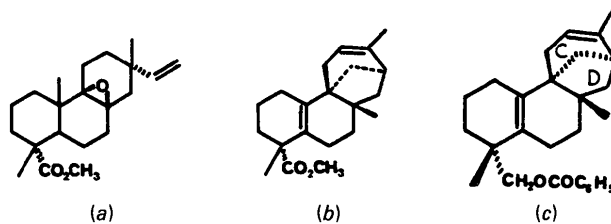*Institut du Pin, Université de Bordeaux I, 33405 Talence CEDEX, France*

**Abstract.** $M_r = 390·6$, monoclinic, $P2_1$, $a = 11·272$ (4), $b = 7·546$ (3), $c = 13·164$ (4) Å, $\beta = 96·94$ (4)°, $V = 1111·5$ (8) Å³, $Z = 2$, $D_x = 1·17$ g cm⁻³... Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 0·77$ cm⁻¹, $F(000) = 424$, $T = 293$ K, $R = 0·065$ for 1230 observed reflections. The title compound is a benzoyloxymethyl derivative of a synthesis intermediate, which could be used for the synthesis of biologically active molecules. The bond lengths and angles are in agreement with those of similar compounds. No short intermolecular contacts exist.

**Introduction.** Lors du réarrangement de l'époxy-8,9β isopimarate de méthyle avec l'éthérate de trifluorure de bore (Taran & Delmond, 1984), nous avons pu mettre en évidence un hydrocarbure diterpénique (*b*). Les analyses physicochimiques (spectrométrie de masse et RMN ¹³C) ont pu montrer que ce composé possédait une structure tétracyclique avec un système bicyclo-[3.2.1]octène au niveau des cycles *C* et *D*.



(*a*) (*b*) (*c*)

Cependant, plusieurs hypothèses structurales étant envisageables, l'analyse radiocristallographique a été entreprise afin de déterminer la conformation tridimensionnelle de ce composé. N'ayant pu obtenir des cristaux de qualité satisfaisante avec ce dérivé, nous avons dû réaliser l'analyse radiocristallographique sur la molécule dérivée (*c*) dans laquelle le groupe méthoxycarbonyl est substitué par le groupe benzoyloxyméthyle.

**Partie expérimentale.** Cristal incolore obtenu dans 2-propanol à 177 K. Dimensions approximatives