

Fig. 1. Bond distances (Å) and angles ( $^{\circ}$ ) with the numbering scheme. E.s.d.'s are in parentheses. Numbering of H atoms is the same as that of the attached carbon atoms.

1.353 (8) Å. In the case of several *N*-alkylphenazinium cations, the ring C—N bond distances adjacent to the alkyl groups are found to be longer than the non-alkylated C—N ring bonds (Morosin, Plastas, Coleman & Stewart, 1978; Endres, Keller, Moroni & Nöthe, 1979; Harms, Keller, Nöthe & Wehe, 1982). Although  $N \cdots I$  is not a covalent bond in this complex, the stronger charge-transfer force about the N(2) atom elongates the N(2)—C(6) distance.

The average deviation of the ring atoms from the least-squares plane of phenazine is 0.02 (2) Å with maximum deviations of 0.05 (2) and 0.04 (2) Å for C(5) and C(2), respectively. The average distance between the planes of the two adjacent phenazine molecules is 3.46 (2) Å. The angle between the phenazine plane and the *c* axis is 56.3 (1) $^{\circ}$ .

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## Methyl (Diphenylmethylimino)acetate *N*-Oxide, $C_{16}H_{15}NO_3$

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**Abstract.**  $M_r = 269.29$ , monoclinic,  $P2_1/a$ ,  $a = 10.142$  (1),  $b = 13.851$  (3),  $c = 9.986$  (1) Å,  $\beta = 99.961$  (9),  $V = 1381.6$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.28$  (1),

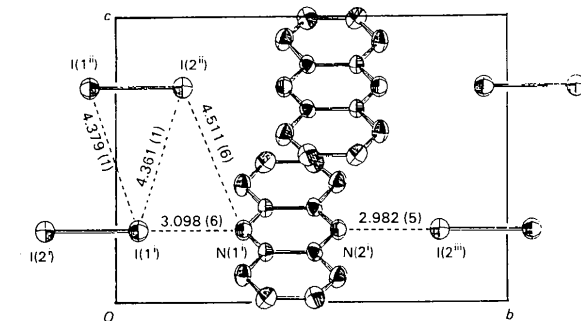


Fig. 2. The packing arrangement projected onto (100), with several intermolecular distances (Å). 50% probability ellipsoids are shown (Johnson, 1965). H atoms have been omitted. Symmetry code: (i)  $x, y, z$ ; (ii)  $-x, -y, 1 - z$ ; (iii)  $x, 1 + y, z$ .

data collection, and also to Mr Tadahisa Noguchi for technical assistance.

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of the C=N bond was determined to be *Z*. The geometry of the molecule conforms to that found in other nitrones.

**Introduction.** As part of the investigation of the chemistry of various *N*-alkyl- $\alpha$ -methoxycarbonyl-methanimine *N*-oxides, a series of geometrical structure determinations has been undertaken (Inouye, Hara & Kakisawa, 1980; Inouye, 1983; Inouye, Takaya & Kakisawa, 1983). The crystal structure of the title compound is reported here.

**Experimental.** Synthesis by the method of Hara, Inouye & Kakisawa (1981), colorless prisms recrystallized from benzene, density measured in heptane-carbon tetrachloride by flotation; systematic absences (from precession photographs)  $0k0$  for  $k$  odd and  $h0l$  for  $h$  odd, space group  $P2_1/a$ ; crystal  $0.38 \times 0.68 \times 0.22$  mm; Rigaku AFC-5 automated four-circle diffractometer; graphite-monochromated  $\text{Mo K}\alpha$  radiation, 40 kV and 180 mA; unit-cell dimensions by least-squares refinement from 25 reflections with  $35 \leq 2\theta \leq 40^\circ$ ;  $2\theta$ - $\omega$  scan, scan rate  $2^\circ \text{ min}^{-1}$ ,  $\Delta\omega = (1.0 + 0.5 \tan\theta)^\circ$ ; three standard reflections measured every 50 reflections with  $\pm 3.5\%$  fluctuation; 4467 unique reflections,  $2\theta = 67^\circ$  ( $0 \leq h \leq 16$ ,  $0 \leq k \leq 22$ ,  $-16 \leq l \leq 16$ ); 2399 reflections with  $|F_o| > 3\sigma(F)$  used in structure determinations; intensities corrected for Lorentz and polarization, not for absorption; structure solved by direct methods (*MULTAN78*, Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); first *E* map revealed position of diphenylmethyl group; successive difference Fourier syntheses determined positions of all non-hydrogen atoms; structure refined on *F* by full-matrix least squares with *RFINE2* (Finger & Prince, 1975),  $R = 0.114$  for anisotropic temperature factors; all H atoms located from difference Fourier synthesis; final refinement with anisotropic temperature factors for all non-hydrogen atoms and isotropic for H atoms,  $R = 0.069$ ,  $R_w = 0.048$ ,  $w = 1/\sigma^2(F)$ ;  $|\Delta\rho|_{\text{max}}$  in final difference Fourier =  $0.36 \text{ e } \text{\AA}^{-3}$ ;  $(\Delta/\sigma)_{\text{max}} = -0.01$ ,  $(\Delta/\sigma)_{\text{av}} = 0.00$ ; all numerical calculations carried out on a FACOM M-200 computer in the Science Information Processing Center of the University of Tsukuba; scattering factors for C, N, O from Cromer & Mann (1968) and for H from *International Tables for X-ray Crystallography* (1974).

**Discussion.** The final atomic parameters are listed in Table 1.\* The molecular structure of the compound is illustrated in Fig. 1, projected on the ester plane

\* Lists of structure factors, anisotropic temperature factors, H-atom coordinates, bond distances involving H atoms, and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38827 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic coordinates of the non-H atoms ( $\times 10^4$ ) and equivalent isotropic temperature factors with e.s.d.'s in parentheses*

	x	y	z	$B_{\text{eq}}^*(\text{\AA}^2)$
C(1)	5006 (5)	6596 (4)	-246 (3)	7.30
O(2)	4860 (2)	6812 (2)	1143 (2)	5.61
C(3)	5726 (2)	7410 (2)	1836 (2)	3.65
O(4)	6625 (2)	7759 (2)	1392 (2)	7.26
C(5)	5400 (2)	7522 (2)	3205 (2)	3.34
N(6)	6294 (2)	7693 (1)	4272 (2)	2.94
O(7)	7550 (1)	7769 (1)	4297 (2)	3.86
C(8)	5844 (2)	7735 (2)	5637 (2)	2.87
C(9)	6324 (2)	8656 (2)	6381 (2)	3.09
C(10)	6430 (3)	8694 (2)	7779 (3)	4.27
C(11)	6705 (3)	9556 (2)	8478 (3)	5.30
C(12)	6899 (3)	10382 (2)	7785 (4)	5.31
C(13)	6824 (3)	10350 (2)	6400 (4)	4.93
C(14)	6534 (2)	9497 (2)	5699 (3)	3.94
C(15)	6224 (2)	6793 (2)	6357 (2)	2.90
C(16)	5261 (2)	6083 (2)	6326 (2)	3.53
C(17)	5592 (3)	5193 (2)	6909 (3)	4.26
C(18)	6883 (3)	4999 (2)	7527 (3)	4.51
C(19)	7845 (3)	5706 (2)	7576 (3)	4.48
C(20)	7522 (2)	6599 (2)	6997 (2)	3.78

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

Table 2. *Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with their e.s.d.'s in parentheses*

C(1)–O(2)	1.452 (4)	C(10)–C(11)	1.387 (4)
O(2)–C(3)	1.313 (3)	C(11)–C(12)	1.369 (5)
C(3)–O(4)	1.184 (3)	C(12)–C(13)	1.373 (5)
C(3)–C(5)	1.470 (3)	C(13)–C(14)	1.379 (4)
C(5)–N(6)	1.296 (3)	C(15)–C(16)	1.382 (3)
N(6)–O(7)	1.274 (2)	C(16)–C(17)	1.379 (4)
N(6)–C(8)	1.511 (3)	C(17)–C(18)	1.374 (4)
C(8)–C(9)	1.514 (3)	C(18)–C(19)	1.377 (4)
C(8)–C(15)	1.507 (3)	C(19)–C(20)	1.381 (4)
C(9)–C(10)	1.382 (4)	C(20)–C(15)	1.387 (3)
C(9)–C(14)	1.384 (3)		
C(1)–O(2)–C(3)	117.6 (3)	C(10)–C(9)–C(14)	118.2 (2)
O(2)–C(3)–O(4)	123.3 (2)	C(10)–C(11)–C(12)	119.9 (3)
O(4)–C(3)–C(5)	128.1 (2)	C(11)–C(12)–C(13)	119.6 (3)
C(3)–C(5)–N(6)	123.1 (2)	C(12)–C(13)–C(14)	120.6 (3)
C(5)–N(6)–O(7)	125.9 (2)	C(13)–C(14)–C(9)	120.6 (3)
O(7)–N(6)–C(8)	115.9 (1)	C(8)–C(15)–C(16)	118.9 (2)
C(5)–N(6)–C(8)	118.1 (2)	C(8)–C(15)–C(20)	122.1 (2)
N(6)–C(8)–C(9)	110.7 (2)	C(16)–C(15)–C(20)	118.8 (2)
N(6)–C(8)–C(15)	107.7 (2)	C(15)–C(16)–C(17)	120.5 (2)
C(9)–C(8)–C(15)	117.5 (2)	C(16)–C(17)–C(18)	120.5 (2)
C(8)–C(9)–C(10)	119.4 (2)	C(17)–C(18)–C(19)	119.4 (2)
C(8)–C(9)–C(14)	122.1 (2)	C(18)–C(19)–C(20)	120.4 (2)
C(9)–C(10)–C(11)	121.0 (2)	C(15)–C(20)–C(19)	120.3 (2)

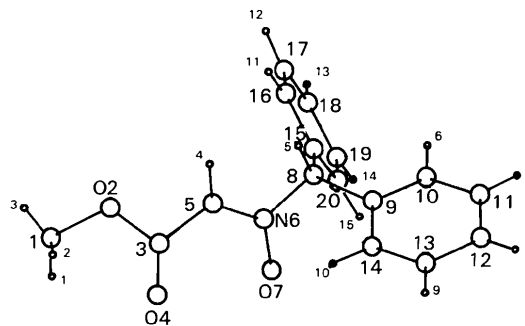


Fig. 1. Molecular structure and numbering. The C atoms are denoted by numerals only. The small circles and numerals indicate the H atoms.

[O(2)–C(3)–O(4)], together with the atomic numbering scheme. The bond distances and angles are listed in Table 2.

The configuration of the C=N bond was proved to be *Z* and the geometry of the nitron chromophore [*R*–CH=N(O)*R'*] was found to be essentially the same as those of other aldonitrone (see Inouye, 1983).

The dihedral angle between the ester plane [O(2)–C(3)–O(4)] and the nitron plane [C(5)–N(6)–O(7)] is 31.0 (3)°, which is larger than that (8.4°) of the *N*-benzyl derivative (Inouye, 1983).

The dihedral angle between the two phenyl rings is 109.2 (3)° and the conformation of the diphenylmethyl group is such that C(5) is eclipsed with H(4) and that C(9) and C(15) atoms are *gauche* to O(7). A similar orientation of phenyl rings was recently reported in phenyl α-[(α-phenylbenzyl)imino]benzyl ketone (Fonseca, Martínez-Carrera & García-Blanco, 1982).

All the intermolecular atomic contacts are slightly larger than usual van der Waals distances; the shortest are O(2) (*x, y, z*)–C(18) (1–*x, 1–y, 1–z*) [3.463 (4) Å] between non-hydrogen atoms, O(2) (*x, y, z*)–H(13)

(1–*x, 1–y, 1–z*) [2.86 (3) Å] involving hydrogen atoms, and H(6) (*x, y, z*)–H(2) (*x, y, z + 1*) [2.39 (5) Å] between hydrogen atoms.

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Acetyltriphenylmethane,\* C<sub>21</sub>H<sub>18</sub>O

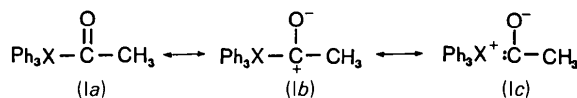
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**Abstract.** *M<sub>r</sub>* = 286.37, monoclinic, *C*2/*c*, *a* = 16.276 (4), *b* = 7.067 (1), *c* = 27.389 (7) Å, β = 99.78 (1)°, *V* = 3104.6 (12) Å<sup>3</sup>, *Z* = 8, *D<sub>m</sub>*(floatation) = 1.209, *D<sub>x</sub>* = 1.225 g cm<sup>-3</sup>, Mo *Kα*, λ = 0.71073 Å, μ = 0.39 cm<sup>-1</sup>, *F*(000) = 1216, room temperature, final *R* = 0.033 for 1086 significant reflections. The acetyl and three phenyl groups are arranged tetrahedrally around the central carbon atom. Two distinct C(central)–C bond lengths are found, C–C(Ph) = 1.538 (2) Å and C–C(Ac) = 1.555 (3) Å, the lengthening of the latter bond being explicable in terms of the electronic structure of the molecule.

**Introduction.** Crystal structure studies of acetyltriphenylgermane (Harrison & Trotter, 1968) and -silane (Chieh & Trotter, 1969) (Ia) (*X* = Ge, Si) have revealed lengthenings of the *X*–C(Ac) bonds, which are explicable in terms of canonical resonance forms, (Ic), and which are in accord with the spectral and basicity studies (Brook, 1957; Brook, Quigley, Peddle, Schwartz & Warner, 1960). The present study shows a



similar but less extreme lengthening in the carbon analogue, (Ia) (*X* = C).

**Experimental.** Colorless crystals, 0.2 × 0.4 × 0.2 mm, CAD-4 diffractometer; graphite-monochromatized Mo *Kα* radiation, θ ≤ 25°, ω–2θ scan, ω-scan width (0.70 + 0.35 tanθ)° extended 25% on each side for background measurement, horizontal aperture (2.00 + tanθ) mm, vertical aperture 4 mm; intensity and orientation controls regularly, < ±0.5% deviation for three reference reflections throughout data collection; cell parameters from least-squares fit on sin<sup>2</sup>θ values for 20 centered reflections with 10 < θ < 18°; Lp corrections (no absorption); 2741 independent reflections, 1086 with *I* ≥ 3σ(*I*) where σ<sup>2</sup>(*I*) = *S* + 2*B* + [0.04(*S* – *B*)]<sup>2</sup>, *S* = the scan count and *B* = the time-averaged background [θ ≤ 20°, 871 of 1457 (59.8%), 20 < θ ≤ 25°, 215 of 1284 (16.7%) observed reflections, θ ≤ 25°, 39.6%]. *MULTAN80* (Main, Fiske, Hull, Lessinger,

\* 1,1,1-Triphenylacetone, 1,1,1-triphenyl-2-propanone.