Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and temperature factors* ($Å^2 \times 10^3$) *with e.s.d.'s in parentheses*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O(1)	5282 (1)	5115 (3)	3144 (4)	44 (1)
N(1)	4108 (2)	3237 (4)	238 (7)	55 (2)
N(2)	4478 (2)	3889 (4)	1181 (7)	45 (2)
N(3)	5709 (2)	5387 (4)	4329 (6)	43 (2)
N(4)	5638 (2)	3315 (4)	4016 (6)	47 (2)
C(1)	3670 (3)	3824 (6)	−625 (8)	43 (3)
C(2)	3296 (3)	3083 (6)	−1560 (8)	53 (2)
C(3)	2867 (3)	3640 (7)	−2467 (9)	64 (3)
C(4)	2788 (2)	4887 (8)	−2468 (10)	66 (3)
C(5)	3158 (3)	5607 (6)	−1536 (9)	56 (3)
C(6)	3598 (2)	5096 (6)	−636 (7)	47 (2)
C(7)	4856 (2)	3255 (5)	1984 (8)	45 (3)
C(8)	5274 (2)	3867 (5)	3051 (8)	42 (2)
C(9)	5893 (2)	4315 (6)	4792 (9)	45 (2)
C(10)	6340 (3)	4134 (6)	6058 (9)	64 (3)

Table 2. *Final interatomic distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

C(1)—C(2)	1·399 (10)	N(2)—C(7)	1·285 (7)
C(2)—C(3)	1·371 (10)	C(7)—C(8)	1·455 (8)
C(3)—C(4)	1·353 (11)	C(8)—O(1)	1·344 (6)
C(4)—C(5)	1·379 (10)	O(1)—N(3)	1·410 (5)
C(5)—C(6)	1·366 (9)	N(3)—C(9)	1·285 (8)
C(6)—C(1)	1·378 (9)	N(4)—C(9)	1·379 (8)
N(1)—C(1)	1·382 (8)	N(4)—C(8)	1·294 (7)
N(1)—N(2)	1·345 (7)	C(9)—C(10)	1·471 (9)

N(3)—O(1)—C(8)	104·8 (3)	C(3)—C(4)—C(5)	118·2 (6)
N(2)—N(1)—C(1)	121·0 (5)	C(4)—C(5)—C(6)	121·9 (6)
N(1)—N(2)—C(7)	116·3 (4)	C(1)—C(6)—C(5)	119·5 (6)
O(1)—N(3)—C(9)	104·2 (4)	N(2)—C(7)—C(8)	120·9 (5)
C(8)—N(4)—C(9)	101·4 (5)	N(4)—C(8)—C(7)	125·8 (5)
N(1)—C(1)—C(6)	123·0 (6)	O(1)—C(8)—C(7)	119·6 (4)
N(1)—C(1)—C(2)	117·8 (6)	O(1)—C(8)—N(4)	114·5 (5)
C(2)—C(1)—C(6)	119·2 (6)	N(3)—C(9)—N(4)	115·1 (5)
C(1)—C(2)—C(3)	119·2 (6)	N(4)—C(9)—C(10)	121·1 (5)
C(2)—C(3)—C(4)	122·0 (6)	N(3)—C(9)—C(10)	123·8 (6)

the hydrazone–azo question has been discussed in detail by Pendergrass, Paul & Curtin (1972); they list literature values of bond lengths expected for the two tautomers: N—N, 1·33–1·38 Å; N=N, 1·23–1·28 Å; N—C(amide), 1·30–1·41 Å; N=C, 1·27–1·29 Å. The

Table 3. *Torsion angles* (°) *with e.s.d.'s in parentheses*

N(2)—N(1)—C(1)—C(2)	−179·0 (6)	C(9)—N(4)—C(8)—O(1)	0·5 (6)
N(2)—N(1)—C(1)—C(6)	2·8 (9)	C(8)—N(4)—C(9)—C(10)	178·8 (6)
C(1)—N(1)—N(2)—C(7)	177·4 (5)	C(9)—N(4)—C(8)—C(7)	−176·3 (6)
N(1)—N(2)—C(7)—C(8)	−179·5 (5)	N(1)—C(1)—C(6)—C(5)	179·2 (6)
N(2)—C(7)—C(8)—O(1)	−2·9 (8)	N(1)—C(1)—C(2)—C(3)	−178·2 (6)
N(2)—C(7)—C(8)—N(4)	173·7 (6)	C(2)—C(1)—C(6)—C(5)	1·1 (9)
N(3)—O(1)—C(8)—N(4)	0·0 (6)	C(6)—C(1)—C(2)—C(3)	0·1 (1·0)
N(3)—O(1)—C(8)—C(7)	177·0 (5)	C(1)—C(2)—C(3)—C(4)	1·0 (1·1)
C(8)—O(1)—N(3)—C(9)	−0·5 (5)	C(2)—C(3)—C(4)—C(5)	0·7 (1·1)
O(1)—N(3)—C(9)—C(10)	−178·8 (5)	C(3)—C(4)—C(5)—C(6)	0·5 (1·1)
O(1)—N(3)—C(9)—N(4)	0·9 (6)	C(4)—C(5)—C(6)—C(1)	−1·4 (1·0)
C(8)—N(4)—C(9)—N(3)	−0·9 (7)		

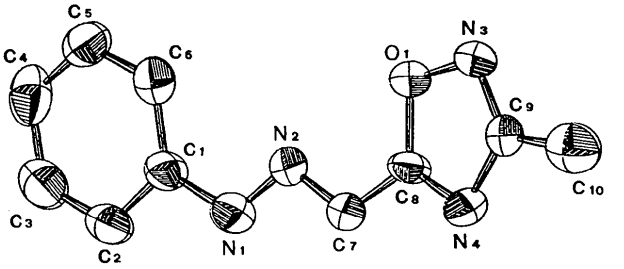

Fig. 1. The molecular structure of the title compound with the atom-numbering scheme.

important point is that our distances fit into the pattern for the hydrazone tautomer. Our N—N distance of 1·345 (7) Å is in the middle of the range quoted while the present N=C distance of 1·285 (7) Å is among the highest values observed in some related compounds.

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3-Isopropyl-2-(4-nitrophenyl)-2,3,4,5-tetrahydro-1,3-oxazine

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Abstract. $C_{13}H_{18}N_2O_3$, $M_r = 250·23$, monoclinic, $P2_1/c$, $a = 11·196$ (2), $b = 16·490$ (3), $c = 7·733$ (1) Å, $\beta = 108·64$ (2)°, $V = 1352·7$ (5) Å^3, $Z = 4$, $D_x = 1·23$ g cm^{-3}, Cu $K\alpha$ radiation, $\lambda = 1·5418$ Å, $\mu = 6·85$ cm^{-1}, $F(000) = 536$, $T = 293$ K, final $R = 0·055$ for 1130 observed reflections. The oxazine ring

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Table 1. *Final fractional coordinates and equivalent isotropic temperature factors for non-H atoms with e.s.d.'s in parentheses*

$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}(Å^2)
O(1)	0·7958 (2)	0·1697 (1)	0·8977 (2)	4·86 (4)
C(1′)	0·9719 (2)	0·1595 (2)	1·1600 (3)	3·70 (6)
N(3)	0·7834 (2)	0·0730 (2)	1·1167 (3)	4·45 (5)
C(2)	0·8295 (2)	0·1534 (2)	1·0871 (3)	4·33 (6)
C(6)	0·6618 (3)	0·1731 (2)	0·8135 (4)	5·78 (8)
C(2′)	1·0295 (3)	0·2205 (2)	1·2791 (4)	4·60 (7)
C(6′)	1·0452 (2)	0·1045 (2)	1·1014 (4)	4·65 (7)
C(3′)	1·1595 (3)	0·2262 (2)	1·3429 (4)	5·21 (8)
C(4′)	1·2299 (2)	0·1698 (2)	1·2875 (4)	4·81 (7)
C(5′)	1·1746 (2)	0·1090 (2)	1·1665 (4)	5·03 (7)
C(4)	0·6452 (3)	0·0723 (2)	1·0394 (4)	6·16 (8)
N(1)	1·3675 (2)	0·1756 (2)	1·3574 (4)	7·48 (8)
C(5)	0·6041 (3)	0·0946 (2)	0·8395 (4)	6·44 (9)
C(7)	0·8324 (3)	0·0452 (2)	1·3087 (4)	5·17 (7)
C(8)	0·8102 (3)	−0·0452 (2)	1·3188 (5)	7·5 (1)
O(2)	1·4301 (2)	0·1251 (2)	1·3126 (4)	11·9 (1)
O(3)	1·4144 (3)	0·2311 (2)	1·4610 (4)	11·8 (1)
C(9)	0·7834 (3)	0·0911 (3)	1·4436 (4)	8·4 (1)

Table 2. *Bond lengths* (Å), *angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

O(1)—C(2)	1·416 (3)	C(6′)—C(5′)	1·375 (4)
O(1)—C(6)	1·432 (3)	C(3′)—C(4′)	1·373 (4)
C(1′)—C(2)	1·514 (3)	C(4′)—C(5′)	1·376 (4)
C(1′)—C(2′)	1·377 (4)	C(4′)—N(1)	1·463 (3)
C(1′)—C(6′)	1·393 (4)	C(4)—C(5)	1·510 (5)
N(3)—C(2)	1·467 (4)	N(1)—O(2)	1·209 (5)
N(3)—C(4)	1·470 (3)	N(1)—O(3)	1·218 (5)
N(3)—C(7)	1·481 (3)	C(7)—C(8)	1·518 (5)
C(6)—C(5)	1·488 (5)	C(7)—C(9)	1·526 (5)
C(2′)—C(3′)	1·381 (4)		

C(2)—O(1)—C(6)	111·8 (2)	C(2′)—C(3′)—C(4′)	119·1 (3)
C(2)—C(1′)—C(2′)	120·7 (2)	C(3′)—C(4′)—C(5′)	121·9 (2)
C(2)—C(1′)—C(6′)	119·7 (2)	C(3′)—C(4′)—N(1)	118·8 (3)
C(2′)—C(1′)—C(6′)	119·6 (2)	C(5′)—C(4′)—N(1)	119·3 (3)
C(2)—N(3)—C(4)	108·7 (2)	C(6′)—C(5′)—C(4′)	118·7 (3)
C(2)—N(3)—C(7)	113·6 (2)	N(3)—C(4)—C(5)	110·5 (3)
C(4)—N(3)—C(7)	114·2 (2)	C(4′)—N(1)—O(2)	119·2 (3)
O(1)—C(2)—C(1′)	105·9 (2)	C(4′)—N(1)—O(3)	118·2 (3)
O(1)—C(2)—N(3)	110·1 (2)	O(2)—N(1)—O(3)	122·6 (3)
C(1′)—C(2)—N(3)	112·5 (2)	C(6)—C(5)—C(4)	110·3 (3)
O(1)—C(6)—C(5)	110·0 (2)	N(3)—C(7)—C(8)	110·0 (2)
C(1′)—C(2′)—C(3′)	120·2 (3)	N(3)—C(7)—C(9)	115·6 (2)
C(1′)—C(6′)—C(5′)	120·5 (2)	C(8)—C(7)—C(9)	110·6 (3)

C(6)—O(1)—C(2)—N(3)	63·3 (3)	C(2)—N(3)—C(4)—C(5)	56·8 (3)
C(2)—O(1)—C(6)—C(5)	−59·1 (3)	O(1)—C(6)—C(5)—C(4)	53·5 (3)
C(4)—N(3)—C(2)—O(1)	−61·3 (3)	N(3)—C(4)—C(5)—C(6)	−53·7 (4)

adopts the chair conformation with the isopropyl group in an equatorial position. The dihedral angle between the planes of the oxazine and phenyl rings is 86·0 (1)°.

Experimental. The title compound (Fig. 1) was prepared by heating an equimolar mixture of 4-nitrobenzaldehyde and 3-isopropylaminopropanol in toluene with azeotropic removal of the water formed. The solvent was then removed by distillation at reduced pressure and the residue crystallized from petroleum ether. Data collected on an Enraf–Nonius CAD-4 diffractometer, graphite monochromator. Crystal dimensions 0·30 × 0·30 × 0·40 mm. Cell parameters measured on the diffractometer using 25 reflections in the 2θ range 20–40°. Range of indices $-12 \le h \le 12$, $0 \le k \le 18$, $0 \le l \le 8$ ($\theta \le 60°$). Three standards (032, 220, 211) measured after every 200 reflections showed a variation of 0·2%. No absorption corrections. Lorentz and polarization corrections. 2015 unique reflections measured. 1130 reflections with $|F_o| > 3\sigma(|F_o|)$. Direct methods (*MULTAN*82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) used for structure determination. H atoms located by difference Fourier synthesis. Anisotropic full-matrix least-squares refinement (on F) for non-H atoms, isotropic for H atoms. In the last cycle the H atoms were fixed at idealized positions (0·94–0·98 Å) with

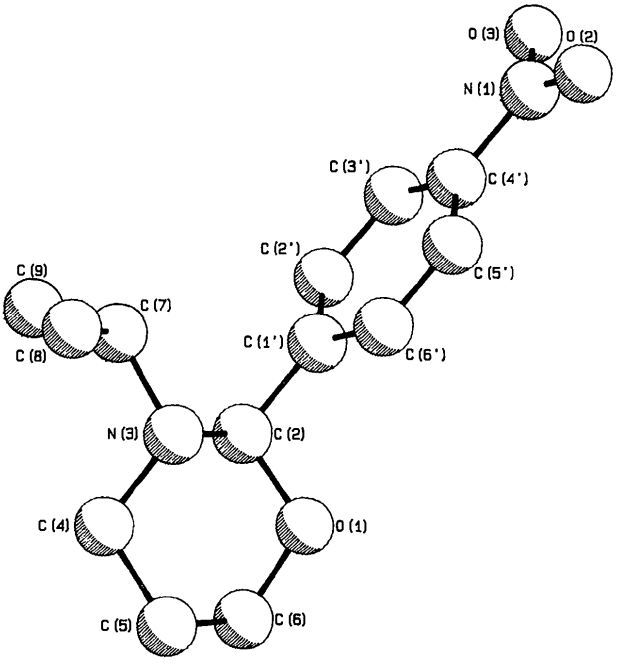

Fig. 1. Numbering of the atoms and conformation of the molecule.

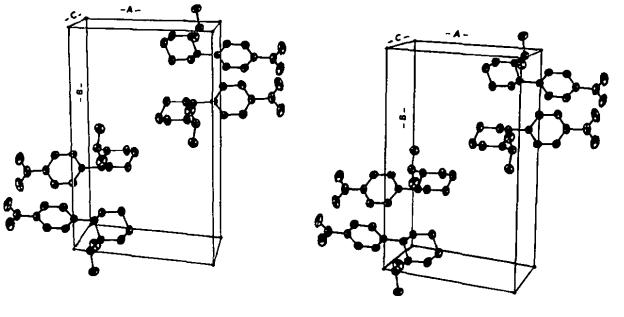

Fig. 2. Stereoview of the unit cell.

fixed Debye–Waller temperature parameters at 6·0 Å^2. $\sum w(|F_o| - |F_c|)^2$ minimized. $w = 4F^2/[\sigma(F)^2 + (pF^2)^2]$, $p = 0{\cdot}04$. $wR = 0{\cdot}055$, max. $\Delta/\sigma = 0{\cdot}02$. Max. peak height in the final difference Fourier map 0·28 e Å^{-3}, $S = 1{\cdot}704$, for 164 variables. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Enraf–Nonius *SDP* (Frenz, 1984). Atomic parameters are given in Table 1;* the bond distances, bond angles, and relevant torsion angles are presented in Table 2. Atomic numbering is shown in Fig. 1, and the packing in Fig. 2.

Related literature. The 4-nitrophenyl group on C(2) is in an equatorial position. The torsion angle for C(6′)—C(1′)—C(2)—O(1) is −64·6 (3)°. Dipole moments and low-temperature NMR studies (Jones, Katritzky & Trepanier, 1971) have also shown that the tetrahydro-1,3-oxazine ring adopts the chair conformation in solution. The solid-state chair conformation is also reported for the tetrahydro-1,2-oxazine systems (Riddell, Murray-Rust & Murray-Rust, 1974).

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53965 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Benzyl 3-Benzyl-3-methyl-2-oxo-5,6-diphenylmorpholin-4-ylcarboxylate

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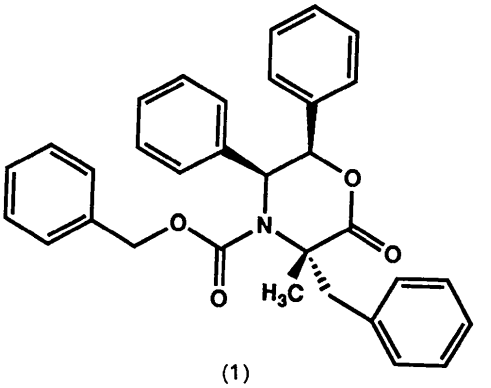
(*Received* 12 *September* 1990; *accepted* 25 *February* 1991)

Abstract. $C_{32}H_{29}NO_4$, $M_r = 491{\cdot}6$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}986$ (1), $b = 15{\cdot}745$ (3), $c = 23{\cdot}633$ (7) Å, $V = 2599{\cdot}5$ (9) Å^3, $Z = 4$, $D_x = 1{\cdot}26$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 6{\cdot}23$ cm^{-1}, $F(000) = 1040$, $T = 115$ K, $R = 0{\cdot}085$ ($wR = 0{\cdot}091$) for 1361 unique, observed reflections. The title compound is disubstituted at the C atom α to the carbonyl C atom.

Experimental. Crystals (colorless prisms) of $C_{32}H_{29}NO_4$ [hereafter (1)] obtained from M. Im and Professor Robert M. Williams (Colorado State University). Crystal size 0·12 × 0·19 × 0·24 mm. Nicolet *R3m* diffractometer, unit-cell constants from least-squares fit of setting angles for 25 reflections ($2\theta_{av} = 43{\cdot}07°$). Data collected ($\theta/2\theta$ scans) to $(\sin\theta)/\lambda = 0{\cdot}5313$ Å^{-1}, $0 \le h \le 8$, $0 \le k \le 17$, $0 \le l \le 26$. Three standard reflections (200, 040, 006) every 97, no change in intensity; Lorentz and polarization corrections; no absorption correction applied; 1918 unique reflections, 1361 reflections with $F_o > 2{\cdot}5\sigma(F_o)$ observed.



(1)

Structure solved by direct methods (*SOLV*) in $P2_12_12_1$; block-diagonal (max. 103 parameters/block, 289 parameters total, data/parameters = 4·7) weighted $\{w = [\sigma^2(F) + gF^2]^{-1}$, $g = 2{\cdot}4 \times 10^{-3}\}$ least-squares refinement on F. H atoms in idealized

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