

fixed Debye–Waller temperature parameters at 6.0 \AA^2 . $\sum w(|F_o| - |F_c|)^2$ minimized. $w = 4F^2/[\sigma(F)^2 + (pF^2)^2]$, $p = 0.04$. $wR = 0.055$, max. $\Delta/\sigma = 0.02$. Max. peak height in the final difference Fourier map 0.28 e \AA^{-3} , $S = 1.704$, 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

* 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.

C(6')—C(1')—C(2)—O(1) is $-64.6(3)^\circ$. 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).

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

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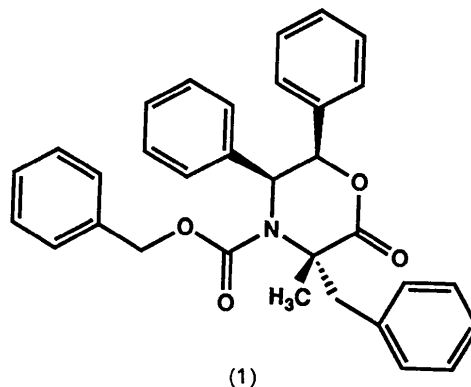
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Abstract. $C_{32}H_{29}NO_4$, $M_r = 491.6$, orthorhombic, $P2_12_12_1$, $a = 6.986(1)$, $b = 15.745(3)$, $c = 23.633(7) \text{ \AA}$, $V = 2599.5(9) \text{ \AA}^3$, $Z = 4$, $D_x = 1.26 \text{ g cm}^{-3}$, $\lambda(\text{Cu } K\alpha) = 1.5418 \text{ \AA}$, $\mu = 6.23 \text{ cm}^{-1}$, $F(000) = 1040$, $T = 115 \text{ K}$, $R = 0.085$ ($wR = 0.091$) 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 $0.12 \times 0.19 \times 0.24 \text{ mm}$. Nicolet $R3m$ diffractometer, unit-cell constants from least-squares fit of setting angles for 25 reflections ($2\theta_{av} = 43.07^\circ$). Data collected ($\theta/2\theta$ scans) to $(\sin \theta)/\lambda = 0.5313 \text{ \AA}^{-1}$, $0 \leq h \leq 8$, $0 \leq k \leq 17$, $0 \leq l \leq 26$. 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 $F_o > 2.5\sigma(F_o)$ observed.



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 $\{w = [\sigma^2(F) + gF^2]^{-1}$, $g = 2.4 \times 10^{-3}\}$ least-squares refinement on F . H atoms in idealized

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Table 1. Atomic coordinates and isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) for (1)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	0.7118 (13)	0.9047 (4)	0.1521 (3)	31 (3)
C(2)	0.6616 (12)	0.8418 (5)	0.1977 (3)	37 (3)
C(3)	0.3485 (14)	0.8214 (7)	0.1571 (4)	64 (4)
C(4)	0.4339 (14)	0.8280 (6)	0.0986 (4)	52 (4)
C(5)	0.4650 (14)	0.7381 (5)	0.0743 (4)	47 (4)
C(6)	0.2776 (15)	0.8733 (7)	0.0627 (4)	73 (5)
C(7)	0.6998 (14)	0.9014 (6)	0.0486 (4)	41 (4)
C(8)	0.9168 (15)	0.9899 (5)	0.0025 (3)	56 (4)
N	0.6149 (10)	0.8786 (4)	0.0990 (3)	38 (3)
O(1)	0.4527 (9)	0.8350 (4)	0.2043 (2)	50 (2)
O(2)	0.1852 (9)	0.7988 (5)	0.1627 (3)	86 (3)
O(3)	0.8447 (9)	0.9555 (4)	0.0552 (2)	38 (2)
O(4)	0.6453 (9)	0.8766 (4)	0.0020 (2)	58 (3)
C(12)	0.8291 (8)	1.0419 (4)	0.1928 (2)	49 (4)
C(13)	0.8064	1.1268	0.2082	65 (5)
C(14)	0.6325	1.1679	0.1984	76 (5)
C(15)	0.4815	1.1240	0.1732	92 (6)
C(16)	0.5042	1.0391	0.1578	68 (5)
C(11)	0.6780	0.9980	0.1676	35 (3)
C(22)	0.6125 (8)	0.9043 (4)	0.2958 (2)	48 (4)
C(23)	0.6832	0.9257	0.3492	68 (5)
C(24)	0.8722	0.9064	0.3634	51 (4)
C(25)	0.9903	0.8658	0.3243	58 (4)
C(26)	0.9196	0.8444	0.2709	48 (4)
C(21)	0.7307	0.8636	0.2567	29 (3)
C(52)	0.5414 (10)	0.6380 (4)	0.1542 (3)	65 (4)
C(53)	0.6722	0.5903	0.1855	78 (6)
C(54)	0.8644	0.5881	0.1695	70 (5)
C(55)	0.9259	0.6337	0.1223	52 (4)
C(56)	0.7951	0.6814	0.0910	42 (3)
C(51)	0.6029	0.6835	0.1069	41 (4)
C(82)	0.9894 (10)	1.1210 (4)	0.0569 (3)	55 (4)
C(83)	1.1057	1.1910	0.0683	67 (5)
C(84)	1.2775	1.2017	0.0391	92 (6)
C(85)	1.3331	1.1424	-0.0016	126 (7)
C(86)	1.2168	1.0724	-0.0130	107 (6)
C(81)	1.0450	1.0618	0.0162	54 (4)

Table 2. Bond lengths (\AA) and angles ($^\circ$) for (1)

C(1)—C(2)	1.505 (11)	C(1)—N	1.483 (10)
C(1)—C(11)	1.533 (10)	C(2)—O(1)	1.471 (11)
C(2)—C(21)	1.516 (9)	C(3)—C(4)	1.511 (13)
C(3)—O(1)	1.348 (11)	C(3)—O(2)	1.202 (12)
C(4)—C(5)	1.542 (13)	C(4)—C(6)	1.556 (14)
C(4)—N	1.495 (12)	C(5)—C(51)	1.503 (11)
C(7)—N	1.378 (11)	C(7)—O(3)	1.332 (11)
C(7)—O(4)	1.229 (11)	C(8)—O(3)	1.447 (10)
C(8)—C(81)	1.478 (12)		
C(2)—C(1)—N	108.5 (6)	C(2)—C(1)—C(11)	115.0 (6)
N—C(1)—C(11)	113.4 (6)	C(1)—C(2)—O(1)	110.8 (7)
C(1)—C(2)—C(21)	115.9 (6)	O(1)—C(2)—C(21)	103.6 (6)
C(4)—C(3)—O(1)	122.2 (8)	C(4)—C(3)—O(2)	119.6 (9)
O(1)—C(3)—O(2)	118.0 (9)	C(3)—C(4)—C(5)	109.4 (8)
C(3)—C(4)—C(6)	104.7 (8)	C(5)—C(4)—C(6)	108.5 (8)
C(3)—C(4)—N	111.4 (7)	C(5)—C(4)—N	111.9 (7)
C(6)—C(4)—N	110.7 (8)	C(4)—C(5)—C(51)	115.1 (7)
N—C(7)—O(3)	113.2 (7)	N—C(7)—O(4)	123.9 (9)
O(3)—C(7)—O(4)	122.9 (8)	O(3)—C(8)—C(81)	108.0 (6)
C(1)—N—C(4)	122.7 (6)	C(1)—N—C(7)	117.5 (7)
C(4)—N—C(7)	119.8 (7)	C(2)—O(1)—C(3)	117.4 (6)
C(7)—O(3)—C(8)	113.8 (6)	C(1)—C(11)—C(12)	117.4 (4)
C(1)—C(11)—C(16)	122.6 (4)	C(2)—C(21)—C(22)	121.7 (4)
C(2)—C(21)—C(26)	118.3 (4)	C(5)—C(51)—C(52)	120.5 (4)
C(5)—C(51)—C(56)	119.5 (4)	C(8)—C(81)—C(82)	119.6 (4)
C(8)—C(81)—C(86)	120.3 (4)		

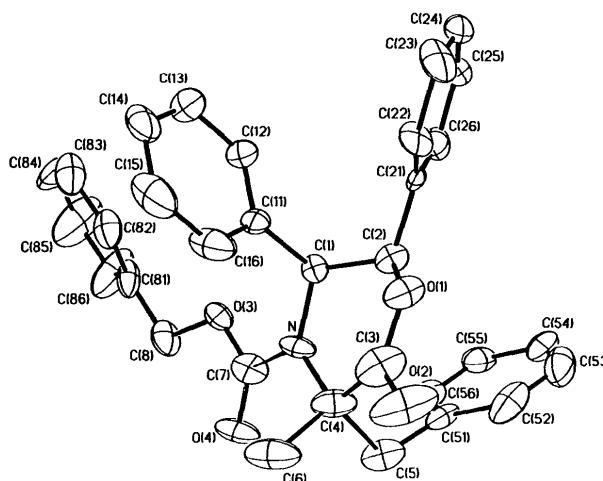


Fig. 1. The structure of (1) (40% probability thermal ellipsoids). H atoms have been omitted for clarity.

positions [C—H = 0.96 \AA , $U(\text{H}) = 1.2 \times U_{iso}(\text{C})$]. All non-H atoms refined with anisotropic thermal parameters. Four phenyl rings modeled as rigid groups (C—C = 1.395 \AA). At convergence [$(\Delta/\sigma)_{\max} = 0.014$, $(\Delta/\sigma)_{\text{mean}} = 0.004$ for last three cycles] $R = 0.085$, $wR = 0.091$, $S = 1.221$, slope of normal probability plot = 1.029, $(\Delta\rho)_{\max} = 0.30$, $(\Delta\rho)_{\min} = -0.35 \text{ e \AA}^{-3}$. Known stereochemistry at C(1)(S) and C(2)(R) (Im, 1990) from synthetic precursor gave relative stereochemistry at C(4)(S) and fixed the enantiomorph. Neutral-atom scattering factors and anomalous-dispersion corrections used (*International Tables for X-ray Crystallography*, 1974, Vol. IV); all calculations performed on a Data General Eclipse S/140 computer using the *SHELXTL* program library (Sheldrick, 1983). Table 1 gives atomic coordinates, and Table 2 gives bond lengths and angles.* Fig. 1 shows the structure of (1), as well as the numbering scheme used.

* Lists of anisotropic thermal parameters, H-atom coordinates and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54034 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. Five compounds that contain a tetrahydro-1,4-oxazin-2-one substructure with an amide N atom have been structurally characterized: (1*S*,4*S*)-*N*-acetyl-3-oxo-5-aza-2-oxabicyclo[2.2.1]heptane (Lenstra, Petit & Geise, 1979), 7,2'-anhydro- β -D-arabinosylorotidine (Smith, Chwang & Sundaralingam, 1980), 3-allyl-4-benzoyloxycarbonyl-5,6-diphenyltetrahydro-1,4-oxazin-2-one (Sinclair, Zhai, Reibenspies & Williams, 1986), desacetyltryptoquinavine *p*-bromophenylurethane (Clardy, Springer, Buchi, Matsuo & Wightman, 1975) and *N*-methyl-3,6-bis(isopropyl)morpholine-2,5-dione (Zhukhlistova, Smirnova, Tishchenko & Andrianov, 1977; Zhukhlistova & Tishchenko, 1980). The title compound (1) differs from these related compounds

in that it is disubstituted at the atom α to the carbonyl C atom.

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Tricyclo[8.2.1.0^{2,9}]trideca-5,11-dien-13-one

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Abstract. C₁₃H₁₆O, $M_r = 188.3$, orthorhombic, $P2_12_12_1$, $a = 8.6255$ (12), $b = 8.8372$ (12), $c = 13.6741$ (7) Å, $V = 1042.3$ (3) Å³, $Z = 4$, $D_x = 1.200$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 5.36$ cm⁻¹, $F(000) = 408$, $T = 297$ K, $R = 0.031$ for 1785 observations with $I > 3\sigma(I)$ (of 2138 unique data). The cyclooctene ring adopts the twist-boat conformation. There is a near-zero torsion angle of the boat at the ring-fusion bond, with magnitude of -2.8 (2)°. The torsion angles about the bonds comprising the sides of the boat are 33.8 (2) and 18.5 (2)°. The cyclooctene and norbornene C=C bonds have lengths of 1.297 (3) and 1.318 (2) Å, respectively, and the C=O bond length is 1.205 (2) Å.

Experimental. The title compound was prepared by allowing one equivalent of 1,1-dimethoxy-2,3,4,5-tetrachlorocyclopentadiene to react with eight equivalents of 1,5-cyclooctadiene neat at 406–413 K (Akhtar, Fray & Yarrow, 1968) followed by reduction in sodium-ethanol and hydrolysis with sulfuric acid-water-ether (Eaton, Sidhu, Langford, Cullison & Pietruszewski, 1987).



Crystals that deposited from the reaction flask, m.p. 338–340 K, were suitable; a clear colorless crystal with dimensions $0.15 \times 0.18 \times 0.32$ mm was used for data collection on an Enraf-Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections having $30 > \theta > 25^\circ$. The ω - 2θ scans were designed for $I = 20\sigma(I)$, subject to max. scan time = 60 s, scan rates varied from 1.0 – 4.1° min⁻¹. A hemisphere of data having $2 < \theta < 75^\circ$, $0 \leq h \leq 10$, $-11 \leq k \leq 11$, $-17 \leq l \leq 17$ was measured and corrected for background, Lorentz, polarization and decay. ψ scans of four reflections exhibited no decrease in intensity with rotation about the diffraction vector, thus no absorption correction was applied. Three standard reflections (400, 031, 006) decreased in intensity by 5.0% apparently due to sublimation and a linear correction was applied. 4637 data were measured, equivalent data were averaged, $R_{\text{int}} = 0.014$, yielding 2138 unique data. Systematic absences $h00$ with h odd, $0k0$ with k odd and $00l$ with l odd indicated space group $P2_12_12_1$. The structure was solved by direct methods using RANTAN (Yao, 1981), refined by full-matrix least squares based upon F , using data for which $I > 3\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using the Enraf-Nonius Structure Determination Package (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), and anomalous coefficients of Cromer (1974). Heavy-atom coordinates were refined with anisotropic thermal parameters; H-atom coordinates were located by ΔF and were refined with

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