Acta Cryst. (1977). **B33**, 594–596

4-(2,4-Dinitro)benzylidene-2-phenyloxazolin-5-one

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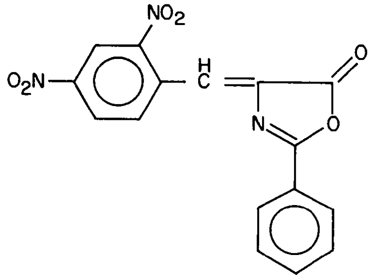
(*Received* 21 *September* 1976; *accepted* 8 *October* 1976)

Abstract. $C_{16}H_9N_3O_6$, F.W. 339·3; monoclinic, $P2_1/n$, $a = 14·309$ (7), $b = 14·453$ (7), $c = 7·107$ (4) Å, $\beta = 92·66$ (5)°, $D_m = 1·533$ g cm^{-3}, $Z = 4$, $D_x = 1·534$ g cm^{-3}. The molecule is approximately planar.

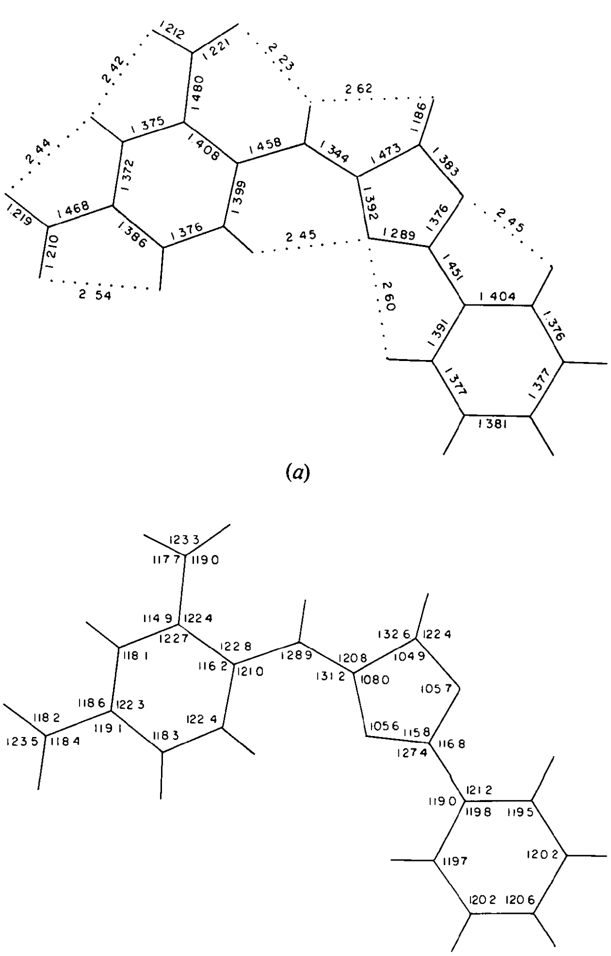
Introduction. It has recently been shown that 4-dimethylamino-3-nitro(α-benzamido)cinnamic acid, when bound to papain, suffers changes such that some spectral features of the acyl-enzyme are characteristic of azlactones (Carey, Carriere, Lynn & Schneider, 1976). The crystal-structure analysis of the azlactone I (the title compound) was undertaken in order to provide conformational data to assist in the interpretation of the resonance Raman spectra and of the reaction mechanism.



(I)

The crystals supplied were orange plates ⟨100⟩, elongated along **c**, with the prominent form ⟨110⟩. The space group was determined from single-crystal photographs (systematic absences: $h0l$ for $h + l$ odd, $0k0$ for k odd). The specimen used for intensity measurements was of dimensions 0·10 × 0·35 × 0·35 mm. This was mounted on a four-circle diffractometer with **b** parallel to the φ axis. The intensities of the 2494 independent reflexions within the limiting sphere $2\theta = 130°$ were measured with Ni-filtered Cu $K\alpha$ radiation and a scintillation counter. The θ–2θ scan mode was used, with scans of 2° for $2\theta < 100°$, and 3° otherwise. Background counts were accumulated for 10 s at the end of each scan. For 257 reflexions the net count was less than either 50, or 10% of the corresponding background, and these were treated as unobserved. Absorption corrections, ranging from 1·10 to 1·38, were applied to the intensities ($\mu = 10·4$ cm^{-1}).

The structure was determined by symbolic addition procedures. Refinement was by block-diagonal least squares, minimizing $\Sigma\, w\Delta F^2$. The H atoms were placed in chemically reasonable positions, and were included in the refinement. The more intense reflexions were judged to suffer from extinction, with intensities reduced to as little as 60% of the calculated value. No correction was applied, but the eight most intense



(*a*)

(*b*)

Fig. 1. (*a*) Some interatomic distances (Å). E.s.d.'s are 0·03 to 0·04 Å where H is involved, and 0·003 to 0·004 Å otherwise. C—H distances range from 0·87 to 0·98 Å. (*b*) Some bond angles (°). E.s.d.'s are 0·2 to 0·3°. The molecule is oriented as in Fig. 2.

reflexions were omitted from the refinement process. Otherwise, the weighting scheme used in the final stages was $w = w_1 w_2$, where $w_1 = F_o/6$ for $F_o < 6$, $w_1 = 6/F_o$ otherwise, and $w_2 = 3{\cdot}14 \sin^2\theta$ for $\sin^2\theta < 0{\cdot}35$, $w_2 = 1$ otherwise. With this weighting scheme there was no obvious dependence of the weighted residual on F_o or θ. On termination of refinement R (for observed reflexions only) was 0·058.* On the final refinement cycle the largest coordinate shift of a non-hydrogen atom was less than 5% of the corresponding e.s.d. of 0·003 Å. The final ΔF synthesis contained no electron density outside the limits $\pm 0{\cdot}2$ e Å^{-3}. The final atomic positions are given in Table 1.*

The computer programs used were those of Ahmed, Hall, Pippy & Huber (1966). Scattering factors were taken from Stewart, Davidson & Simpson (1965) for H, and from Hanson, Herman, Lea & Skillman (1964).

Table 1. *Final atomic coordinates* (× 10^3 *for* H *atoms,* × 10^4 *otherwise*)

E.s.d.'s are given in parentheses.

	x	*y*	*z*
O(1)	4246 (1)	11131 (1)	1697 (3)
C(2)	5015 (2)	10659 (1)	2429 (3)
N(3)	4942 (1)	9770 (1)	2457 (3)
C(4)	4048 (2)	9576 (1)	1705 (3)
C(5)	3590 (2)	10461 (2)	1200 (4)
O(6)	2839 (1)	10645 (1)	528 (4)
C(7)	3600 (1)	8768 (1)	1408 (3)
C(8)	3937 (2)	7830 (1)	1780 (3)
C(9)	3383 (2)	7040 (2)	1408 (3)
C(10)	3724 (2)	6155 (2)	1624 (4)
C(11)	4631 (2)	6046 (2)	2300 (3)
C(12)	5205 (2)	6792 (2)	2759 (4)
C(13)	4856 (2)	7670 (2)	2457 (3)
N(14)	2386 (1)	7101 (1)	769 (3)
O(15)	1922 (1)	7756 (1)	1277 (3)
O(16)	2065 (2)	6476 (2)	−196 (3)
N(17)	5004 (2)	5105 (1)	2535 (4)
O(18)	4531 (2)	4463 (1)	1916 (4)
O(19)	5767 (2)	5009 (2)	3315 (5)
C(20)	5821 (2)	11211 (1)	3030 (3)
C(21)	5784 (2)	12182 (2)	3017 (3)
C(22)	6573 (2)	12681 (2)	3539 (4)
C(23)	7390 (2)	12232 (2)	4079 (4)
C(24)	7431 (2)	11278 (2)	4099 (4)
C(25)	6650 (2)	10764 (2)	3585 (4)
H(7)	300 (2)	885 (2)	92 (3)
H(10)	337 (2)	568 (2)	139 (5)
H(12)	586 (2)	676 (2)	325 (4)
H(13)	522 (2)	810 (2)	278 (3)
H(21)	521 (2)	1246 (2)	261 (3)
H(22)	656 (2)	1334 (3)	352 (5)
H(23)	790 (2)	1257 (3)	447 (5)
H(24)	802 (2)	1099 (2)	437 (4)
H(25)	666 (2)	1010 (2)	364 (4)

Discussion. Bond lengths and angles are given in Fig. 1, and are consistent with the formulation given above. Details of some mean planes are given in Table 2. The oxazolin-5-one system is precisely planar, as is the at-

Table 2. *Distances* (Å × 10^3) *of some atoms from certain mean planes*

Atoms given in bold type define the plane.

1. **O(1)**, 102; **C(2)**, −6; **N(3)**, −42; **C(4)**, 33; **C(5)**, 132; **O(6)**, 216; **C(7)**, 32; **C(8)**, −35; **C(9)**, −37; **C(10)**, 2; **C(11)**, −9; **C(12)**, −55; **C(13)**, −40; **N(14)**, −94; **O(15)**, −679; **O(16)**, 416; **N(17)**, 34; **O(18)**, 235; **O(19)**, −118; **C(20)**, −30; **C(21)**, −60; **C(22)**, −39; **C(23)**, 4; **C(24)**, 31; **C(25)**, 8
2. **C(8)**, 6; **C(9)**, −17; **C(10)**, 9; **C(11)**, 7; **C(12)**, −17; **C(13)**, 10; N(14), −85; O(15), −663; O(16), 407; N(17), 38; O(18), 221; O(19), −106; C(7), 87
3. **O(1)**, 1; **C(2)**, −2; **N(3)**, 3; **C(4)**, −2; **C(5)**, 0; **O(6)**, −3; C(7), −4; C(20), 25
4. C(2), 45; **C(20)**, 2; **C(21)**, −1; **C(22)**, 0; **C(23)**, 0; **C(24)**, 1; **C(25)**, −2

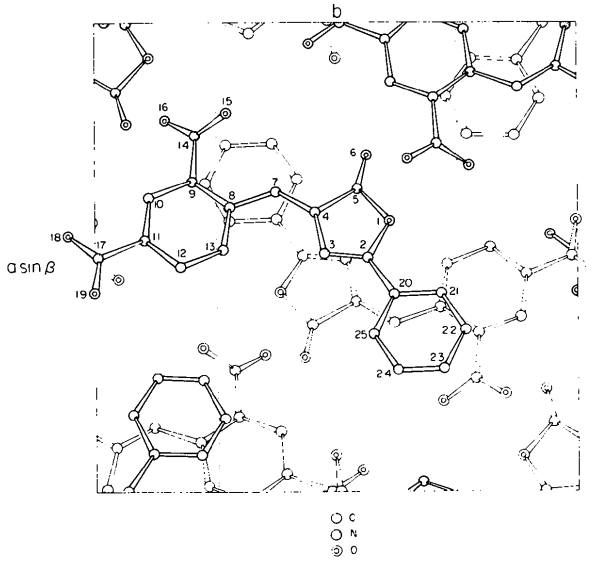


Fig. 2. The structure viewed along **c**. H atoms have been omitted.

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* A list of atomic thermal-motion parameters (anisotropic for the non-hydrogen atoms) and structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32204 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

tached phenyl ring. The six-membered ring of the benzylidene system is nearly planar, and the nitro groups are bent and twisted out of the plane by 30·4 and 9·0°. The molecule as a whole is approximately planar, with no atoms except O atoms of the nitro groups lying more than 0·22 Å from the mean plane. This planarity presumably results from the effects of conjugation; there are some rather short intramolecular N · · · H and O · · · H contacts [2·23 Å for O(15) · · · H(7)], but the distribution of bond angles is consistent with repulsion at the shorter contacts. Molecules related by the centres of symmetry at $\frac{1}{2}$,0,0 and $\frac{1}{2}$,0,$\frac{1}{2}$ overlap each other to form stacks parallel to **c**, with a mean interplanar spacing of 3·39 Å (Fig. 2). The intermolecular distances are consistent with van der Waals interactions.

The author is grateful to Dr Henry Schneider for suggesting this problem and for supplying the specimen material.

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Acta Cryst. (1977). **B33**, 596–599

1-{1-[4,4-Bis(4-fluorophenyl)butyl]-4-piperidinyl}-5-chloro-1,3-dihydro-2*H*-benzimidazol-2-one: Clopimozide

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(*Received* 2 *October* 1976; *accepted* 16 *October* 1976)

Abstract. $C_{28}H_{28}N_3OF_2Cl$, monoclinic, $C2/c$; $a = 21{\cdot}204\,(5)$, $b = 9{\cdot}974\,(5)$, $c = 25{\cdot}906\,(5)$ Å, $\beta = 112{\cdot}26\,(5)°$; $Z = 8$; $D_m = 1{\cdot}31$, $D_c = 1{\cdot}306$ g cm^{-3}.

Introduction. Clopimozide is a neuroleptic of the diphenylbutylamine class.

The space group and lattice parameters were determined on a Nonius CAD-4 four-circle computer-controlled diffractometer. The instrumental settings are given in Table 1.

Table 1. *Instrumental settings for the data collection*

Source: Cu $K\bar{\alpha}$; $\lambda = 1{\cdot}54178$ Å
Scan: ω–2θ
Graphite monochromator
$72° \geq \theta \geq 2°$
$\Delta 2\theta = 0{\cdot}70 + 0{\cdot}3 \operatorname{tg} \theta$
Aperture $= 2{\cdot}5 + 0{\cdot}5 \operatorname{tg} \theta$
Confidence level: $2{\cdot}5\sigma$, where $\sigma^2 = S + B + (0{\cdot}035)^2$, S being the scan and B the background count
Total number of independent reflexions: 4945
Total observed: 2913

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971). Refinement was with the X-RAY72 system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). The coordinates of the H atoms were calculated. With anisotropic temperature factors for all non-H atoms and isotropic ones for H, the final R was 0·08. The scattering factors were those of Cromer & Mann (1968) except for H for which those of Stewart, Davidson & Simpson (1968) were used. The final coordinates and thermal parameters are listed in Table 2. Fig. 1 gives the atomic numbering. Intramolecular bond distances and angles are listed in Table 3.*

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32212 (42 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.