ring, 1·777 (2) Å. This difference suggests that 3*d* orbitals on the P atom may interact to a small extent with the π system of the pyrazole ring. As suggested by Cobbledick & Einstein (1975) the small but significant differences between C—C bonds [0·042 (7) Å] and the two C—N bonds [0·017 (6) Å] in the pyrazole ring support this view. The P—O bond is 1·484 (2) Å which makes the coordination tetrahedra more distorted. The C—C distances in the isopropyl group are about 1·51 Å, which is normal for single bonds. The two phenyl and the pyrazole rings are planar with small deviations and the phenyl-ring planes are orthogonal.

The C—C distances in the phenyl rings are in the range 1·362 (5)–1·394 (4) Å as expected for phenyl (Cobbledick & Einstein, 1975; Rodulfo de Gil, Valentina Rivera & Noguera, 1977).

We are indebted to Dr C. J. Gilmore for helpful discussion.

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Acta Cryst. (1988). C**44**, 294–296

Structure of 2,5-Dihydro-4-[(4-methylphenyl)amino]-2-oxo-*N*-phenyl-3-furancarboxamide

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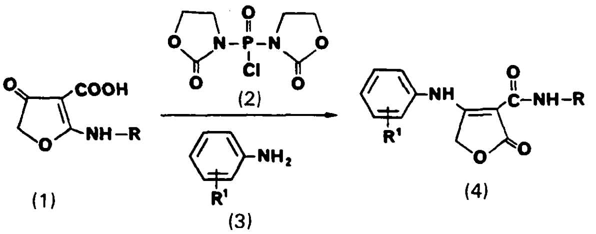
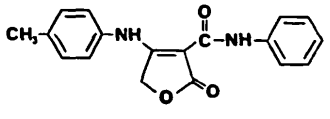
(*Received* 6 *March* 1987; *accepted* 21 *September* 1987)

Abstract. $C_{18}H_{16}N_2O_3$, $M_r = 308{\cdot}34$, monoclinic, $C2/c$, $a = 19{\cdot}482\,(3)$, $b = 7{\cdot}642\,(1)$, $c = 21{\cdot}695\,(3)$ Å, $\beta = 110{\cdot}87\,(1)°$, $V = 3018{\cdot}1\,(8)$ Å^3, $Z = 8$, $D_x = 1{\cdot}357$ g cm^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}88$ cm^{-1}, $F(000) = 1296$, $T = 297$ K, final $R = 0{\cdot}045$ for 1856 unique observed reflections. The title compound was derived from a 4,5-dihydro-4-oxo-2-(phenylamino)-3-furancarboxylic acid *via* a novel 3(2*H*)-furanone–2(5*H*)-furanone rearrangement. The amide and amine N atoms participate in the conjugated π system and the N—H's participate in intramolecular hydrogen bonding.

Introduction. Recently (Mack & Georgiev, 1987), we reported a novel 3(2*H*)-furanone–2(5*H*)-furanone rearrangement which was accomplished by treating 2-(*N*-substituted amino)-4,5-dihydro-4-oxo-3-furancarboxylic acids (1) with 1 equivalent of di(2-oxo-1,3-oxazolidin-3-yl)phosphinic chloride (2) and an appropriately substituted aromatic amine (3), in the presence of triethylamine. The rearrangement resulted in the facile synthesis of a new class of 2(5*H*)-furanone amides (4). An X-ray crystal-structure determination of 2,5-dihydro-4-[(4-methylphenyl)amino]-2-oxo-*N*-phenyl-3-furancarboxamide (4*a*) was undertaken to define unambiguously the structures of 2(5*H*)-furanone amides (4).





(4*a*)

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Table 1. *Refined positional parameters*

$$B_{eq} = \tfrac{1}{3}\sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	B_{eq}/B_{iso}(Å^2)
C(1)	0·5677 (1)	0·9267 (3)	1·1030 (1)	4·50 (5)
C(2)	0·5248 (1)	0·9103 (3)	1·1420 (1)	4·65 (5)
C(3)	0·4650 (1)	0·7998 (3)	1·12523 (9)	4·13 (5)
C(4)	0·4483 (1)	0·7055 (3)	1·0674 (1)	4·29 (5)
C(5)	0·4896 (1)	0·7219 (3)	1·02785 (9)	4·16 (5)
C(6)	0·5502 (1)	0·8309 (3)	1·04563 (9)	3·65 (5)
C(7)	0·6509 (1)	0·9153 (3)	1·00468 (9)	3·60 (5)
C(8)	0·6825 (1)	0·9016 (3)	0·95760 (9)	3·69 (5)
C(9)	0·7496 (1)	0·9988 (3)	0·97934 (9)	4·30 (5)
C(10)	0·6982 (1)	1·0309 (3)	1·05848 (9)	4·15 (5)
C(11)	0·6513 (1)	0·8033 (3)	0·8964 (1)	4·17 (5)
C(12)	0·6729 (1)	0·7381 (3)	0·79171 (9)	4·04 (5)
C(13)	0·6042 (1)	0·6720 (4)	0·7556 (1)	4·92 (6)
C(14)	0·5906 (1)	0·6086 (4)	0·6926 (1)	5·43 (6)
C(15)	0·6435 (1)	0·6108 (3)	0·6649 (1)	5·21 (6)
C(16)	0·7117 (1)	0·6767 (4)	0·7008 (1)	5·15 (5)
C(17)	0·7270 (1)	0·7400 (3)	0·7638 (1)	4·54 (5)
C(18)	0·4187 (1)	0·7807 (4)	1·1677 (1)	5·73 (6)
N(1)	0·58860 (8)	0·8377 (2)	1·00129 (8)	4·08 (4)
N(2)	0·69076 (8)	0·8089 (3)	0·85563 (8)	4·25 (4)
O(1)	0·75969 (7)	1·0718 (2)	1·03935 (6)	5·01 (4)
O(2)	0·59312 (7)	0·7226 (2)	0·88337 (7)	5·28 (4)
O(3)	0·79476 (7)	1·0194 (3)	0·95321 (7)	5·44 (4)
H(N1)	0·571 (1)	0·771 (3)	0·9641 (9)	5·4 (5)
H(N2)	0·7303 (9)	0·868 (3)	0·8709 (9)	4·6 (5)
H(1)	0·6063 (9)	1·008 (3)	1·1158 (9)	5·1 (5)
H(2)	0·536 (1)	0·980 (3)	1·1830 (9)	6·1 (5)
H(4)	0·406 (1)	0·634 (3)	1·0532 (9)	5·1 (5)
H(5)	0·4768 (9)	0·660 (3)	0·9877 (8)	4·4 (5)
H(10)	0·7161 (9)	0·977 (3)	1·1016 (8)	4·1 (4)
H(10′)	0·6739 (9)	1·147 (3)	1·0623 (9)	4·8 (5)
H(13)	0·567 (1)	0·668 (3)	0·774 (1)	6·0 (5)
H(14)	0·544 (1)	0·563 (3)	0·669 (1)	6·2 (6)
H(15)	0·635 (1)	0·566 (3)	0·6198 (9)	5·7 (5)
H(16)	0·751 (1)	0·682 (3)	0·6833 (9)	5·1 (5)
H(17)	0·7711 (9)	0·787 (3)	0·7888 (9)	4·5 (5)
H(18)	0·433 (1)	0·689 (4)	1·197 (1)	10·1 (8)
H(18′)	0·421 (1)	0·880 (4)	1·194 (1)	10·0 (8)
H(18″)	0·372 (1)	0·770 (4)	1·144 (1)	9·7 (8)

H atoms were refined isotropically.

Experimental. Clear colorless crystal (0·25 × 0·20 × 0·40 mm); Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters from 22 reflections with $21 < 2\theta < 28°$. 2967 reflections measured using ω–2θ scan technique within ranges $4 \leq 2\theta \leq 50°$, $0 \leq h \leq 25$, $-9 \leq k \leq 0$, $-28 \leq l \leq 28$, $h + k = 2n$. Intensities of three standard reflections ($80\bar{8}$, $1\bar{1}7$, $1\bar{3}2$) recorded every 2500 s of X-ray exposure showed no significant decay. 2967 reflections measured, 1856 unique observed reflections [$I > 3\sigma(I)$], $R_{int} = 4·1\%$. Data corrected for Lorentz and polarization effects, not for absorption. Structure solved by *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) in the non-centrosymmetric space group *Cc*, which revealed two independent molecules related by a center of symmetry. Thus, subsequent refinement was performed in the centrosymmetric space group *C*2/*c*. H atoms found from subsequent difference Fourier syntheses. Refinement by full-matrix least squares to minimize $w(|F_o - F_c|^2)$ led to $R = 0·045$ and $wR = 0·054$ for 272 variables with $w = 1/\sigma^2(F_o)$ and $S = 1·49$. Non-H atoms were refined with anisotropic thermal parameters; H atoms were refined isotropically. Maximum least-squares-shift-to-e.s.d. ratio 0·02 in final refinement cycle. Largest residual electron density in final difference Fourier synthesis 0·20 e Å^{-3}. Atomic scattering factors from Cromer & Waber (1974); anomalous-dispersion terms from Ibers & Hamilton (1964). All computer programs from Enraf–Nonius *SDP* package (Frenz, 1978).

Discussion. Final positional parameters and equivalent isotropic thermal parameters are given in Table 1.* An *ORTEP* representation of the molecule appears in Fig. 1 and an *ORTEP* drawing of the unit cell in Fig. 2.

The plane defined by N(1), C(7), C(8), C(11), O(2), N(2) is planar within 0·008 Å. The shortening of the N(1)–C(7) bond, 1·328 (3) Å, relative to N(1)–C(6), 1·415 (3) Å, and of the N(2)–C(11) bond, 1·363 (3) Å, relative to N(2)–C(12), 1·412 (3) Å, indicates substantial electron delocalization through the N(1)–C(7)–C(8)–C(11)[–O(2)]–N(2) π system.

Both H(N1) and H(N2) apparently participate in intramolecular hydrogen bonding with O(2) and O(3), respectively: H(N1)···O(2), 1·99 (2), N1···O2, 2·736 (2), H(N2)···O(3), 2·12 (2), N2···O3,

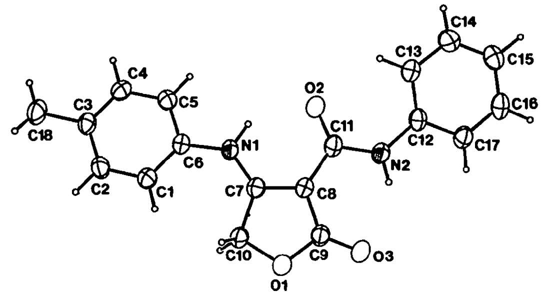

Fig. 1. *ORTEP* (Johnson, 1965) drawing (30% probability thermal ellipsoids) showing atom-numbering scheme.

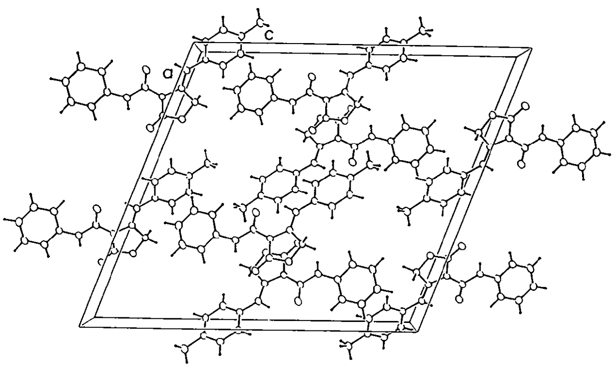

Fig. 2. *ORTEP* view of unit cell along *b* axis.

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* Lists of structure factors, anisotropic thermal parameters and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44390 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

2·849 (2) Å. The O(2), C(11), C(8), C(7), N(1), H(N1) moiety is planar within 0·03 Å and the O(3), C(9), C(8), C(11), N(2), H(N2) moiety is planar within 0·01 Å. There is no evidence of intermolecular hydrogen bonding.

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Acta Cryst. (1988). C**44**, 296–300

Synthesis of Cedranoid Sesquiterpenes. IV. X-ray Characterization of the *endo*-6-Hydroxy-*endo*-8-methyl and *exo*-6-Hydroxy-*exo*-8-methyl Isomers of Methyl 6-Hydroxy-4,4,8-trimethyl-3-oxo-*cis*-bicyclo[3.3.0]octane-1-carboxylate

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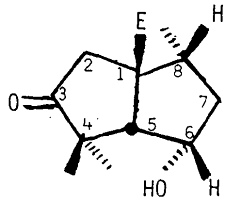
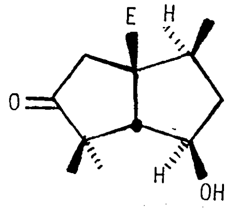
(*Received* 26 *February* 1987; *accepted* 29 *September* 1987)

Abstract. $C_{13}H_{20}O_4$, *endo-endo* isomer (1), $M_r = 240·3$, monoclinic, $P2/n$, $a = 15·041$ (3), $b = 6·225$ (3), $c = 27·795$ (5) Å, $\beta = 101·56$ (2)°, $U = 2550$ (2) Å^3, $Z = 8$ (2 independent molecules), $D_x = 1·25$ g cm^{-3}, Mo $K\bar{\alpha}$ radiation, $\lambda = 0·71069$ Å, $\mu = 0·9$ cm^{-1}, $F(000) = 1040$, $T = 298$ K, $R = 0·090$, $wR = 0·071$ for 1499 reflections with $I \geq 2\sigma(I)$. *exo-exo* Isomer (2), $M_r = 240·3$, rhombohedral, $R\bar{3}$, $a = 12·916$ (7) Å, $\alpha = 92·86$ (5)°, $U = 2146$ (4) Å^3, $Z = 6$, $D_x = 1·12$ g cm^{-3}, Mo $K\bar{\alpha}$ radiation, $\mu = 0·8$ cm^{-1}, $F(000) = 780$, $T = 298$ K, $R = 0·062$, $wR = 0·064$ for 1306 reflections with $I \geq 3\sigma(I)$. In isomer (1), the two independent molecules in the asymmetric unit are approximate mirror images and the 6-hydroxy and 8-methyl substituents are both *endo*. The cyclopentane rings are both flattened envelopes and there are significant transannular O=C···OH contacts (2·88 and 2·91 Å) in both molecules. In isomer (2), the C(6) and C(8) substituents are both *exo* and the cyclopentane rings have distorted envelope and half-chair conformations. The molecules of this isomer are linked by hydrogen bonding into hexameric units.

Introduction. Synthetic routes to the cedranoid sesquiterpenes being pursued in these laboratories (Yates, Burnell, Freer & Sawyer, 1987; Grewal, Hayes, Sawyer & Yates, 1987) have involved the preparation of several bicyclo[3.3.0]octanes as intermediates. In this paper we report on the determination of the relative configurations in two such intermediates, (1) and (2), by X-ray crystallography.



(1)



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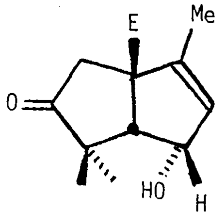

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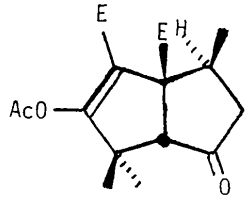

(4)

($E = CO_2Me$)

Experimental. Isomer (1) was prepared by hydrogenation of (3), as described elsewhere (Yates, Burnell, Freer & Sawyer, 1987). Crystallization from pentane containing 5–10% dichloromethane gave colorless crystals as small thin needles, m.p. 395·6–396·1 K. Crystal of overall dimensions 0·019 × 0·069 ×

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