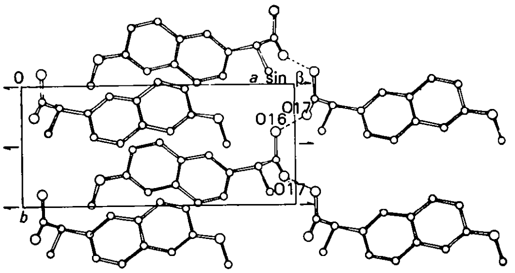

Fig. 3. Packing diagram down the *c* axis.

The rotation of the carboxyl group with respect to the benzene ring in the naphthalene moiety which seems to be connected with the anti-inflammatory potential activity is similar to that of the other 2-substituted propionic acids. The torsion angles τ_3[C(9)–C(8)–C(13)–C(15)] = 70·5 (8)° and τ_4[C(8)–C(13)–C(15)–O(16)(H)] = 90·3 (8)° are comparable with those in other 2-substituted propionic acids [ibuprofen: $\tau_3 = 96{\cdot}4$ (2), $\tau_4 = 89{\cdot}3$ (2)° (McConnel, 1974); flurbiprofen: $\tau_3 = 107{\cdot}7$ (6), $\tau_4 = 77{\cdot}3$ (6)° (Flippen & Gilardi, 1975)]. The theoretical values for the minimum-energy configuration of 2-phenylpropionic acid calculated using CNDO/2 and STD 3G are τ_3 ±60, ±120, τ_4 ±90° (Dupont, Dideberg, Dive, Godfroid & Steiner, 1982).

The packing of the molecules down the *c* axis is shown in Fig. 3. The structure is stabilized by the intermolecular hydrogen bond between O(16) and O(17) of a neighbouring molecule at $(2-x,\ y-\frac{1}{2},\ 2-z)$: O(16)–H(O16) = 1·04 (3), O(16)···O(17) = 2·636 (8) Å, O(16)–H(O16)···O(17) = 163 (3)°.

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Acta Cryst. (1985). C**41**, 282–284

(*Z*)-3-(α-Hydroxybenzylidene)-1,2,3,9-tetrahydropyrrolo[2,1-*b*]quinazolin-9-one, $C_{18}H_{14}N_2O_2$

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Abstract. $M_r = 290{\cdot}3288$, monoclinic, $P2_1/c$, $a = 11{\cdot}309$ (5), $b = 11{\cdot}121$ (5), $c = 11{\cdot}672$ (5) Å, $\beta = 110{\cdot}23$ (5)°, $U = 1377{\cdot}34$ Å^3, $Z = 4$, $D_x = 1{\cdot}4002$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}054$ mm^{-1}, $F(000) = 608$, $T = 293$ K, $R = 0{\cdot}0689$ for 1115 observed reflexions. The hydroxyl proton is involved in hydrogen bonding to the sp^2-hybridized N atom of the quinazoline ring. The phenyl group is close to coplanar with the pyrroloquinazolinone fragment giving the maximum extended π bonding.

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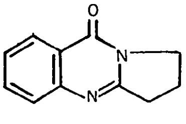
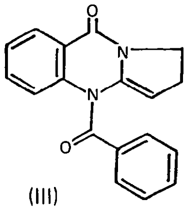
Introduction. Previously we have shown that 1,2,3,9-tetrahydropyrrolo[2,1-*b*]quinazolin-9-one (deoxyvasicione), (I), undergoes condensation at the 3-position of the pyrrolo ring with electrophiles (Dunn, Guy & Kinnear, 1983; Dunn & Kinnear, 1984). Reaction of (I) with benzoic anhydride at 503 K gave the expected enol benzoate along with another compound of spectral mass 290·3288 which could not be identified confidently from the spectroscopic data. These possibilities for the structure were proposed (II, III, IV) and X-ray structural analysis has now shown the product to be (II).


(I)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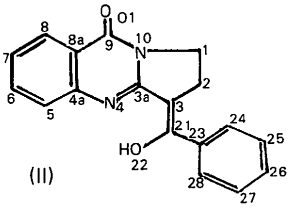
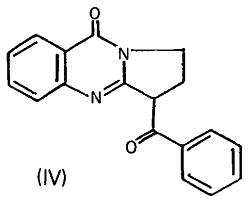


(II)


(IV)

Experimental. The title compound was isolated as the minor product from condensation between 1,2,3,9-tetrahydropyrrolo[2,1-*b*]quinazolin-9-one and benzoic anhydride. Full details of its preparation will be reported elsewhere.

Yellow prisms from dimethylformamide or ethanol, crystals ~0·3 × 0·4 × 0·3 mm, Stoe STADI-2 diffractometer, cell dimensions from zero-level Weissenberg photographs refined on diffractometer. Intensities collected over the range $0 < 2\theta < 55°$ for crystals mounted on the a ($h = 0$ to 2) and b ($k = 0$ to 8) axes. ω-scan technique, 2552 unique reflexions measured of which 1115 with $F_o > 5\sigma(F_o)$ were used in refinement. 2% variation in 2 standard reflexions during data collection. Best E map produced by the routine *TANG* revealed all the non-hydrogen atoms. Atomic coordinates with isotropic thermal parameters were refined on F to $R = 0{\cdot}11$ after which all atoms except H were refined using anisotropic thermal parameters. H atoms were included as riding atoms at calculated positions with grouped isotropic temperature parameters except for H(221) which is involved in hydrogen bonding. This atom was located on a difference Fourier map and refined with an isotropic temperature parameter. It did not refine completely satisfactorily giving a final temperature parameter of 0·16 (3) Å². Final refinement: 216 parameters, $wR = 0{\cdot}065$, $w = 2{\cdot}478/[\sigma^2(F) + 0{\cdot}001305F^2]$, max. shift/e.s.d. = 0·077 [except for H(221)], mean shift/e.s.d. = 0·0166, highest residual peak = 0·26 e Å^{-3}. Atomic scattering factors as in *SHELX*. All calculations performed on the Dundee University DEC-10 computer using *SHELX*76 (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978).

Table 1. *Fractional coordinates* (×10⁴) *and equivalent isotropic thermal parameters* (Å² ×10³)

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

	x	y	z	U_{eq} or U
C(1)	4700 (4)	5088 (4)	8188 (4)	53 (1)
C(2)	5570 (4)	4911 (4)	7437 (4)	49 (1)
C(3)	5001 (4)	3871 (3)	6593 (4)	38 (1)
C(3a)	3887 (4)	3493 (3)	6797 (3)	36 (1)
C(4a)	2085 (4)	2414 (4)	6557 (4)	41 (1)
C(5)	1285 (5)	1472 (4)	5983 (4)	50 (1)
C(6)	206 (5)	1259 (5)	6256 (4)	60 (2)
C(7)	−82 (5)	2001 (5)	7095 (5)	60 (2)
C(8)	709 (5)	2916 (5)	7668 (4)	55 (2)
C(8a)	1810 (5)	3132 (4)	7420 (4)	43 (1)
C(9)	2675 (5)	4078 (4)	8063 (4)	48 (2)
C(21)	5360 (5)	3337 (4)	5709 (4)	39 (1)
C(23)	6470 (5)	3610 (4)	5375 (4)	43 (1)
C(24)	6743 (5)	2839 (5)	4559 (5)	64 (2)
C(25)	7728 (6)	3063 (6)	4160 (5)	77 (2)
C(26)	8482 (5)	4048 (6)	4580 (5)	68 (2)
C(27)	8232 (5)	4817 (5)	5389 (5)	64 (2)
C(28)	7238 (5)	4599 (4)	5790 (4)	55 (2)
N(4)	3146 (3)	2617 (3)	6249 (3)	41 (1)
N(10)	3696 (4)	4196 (3)	7687 (3)	41 (1)
O(01)	2548 (3)	4748 (3)	8847 (3)	68 (1)
O(22)	4634 (3)	2457 (3)	5016 (3)	48 (1)

Table 2. *Interatomic distances* (Å) *and angles* (°)

C(2)–C(1)	1·540 (8)	N(4)–C(3a)–C(3)	126·6 (4)
N(10)–C(1)	1·468 (5)	N(10)–C(3a)–C(3)	109·7 (3)
C(3)–C(2)	1·511 (6)	N(10)–C(3a)–N(4)	123·7 (5)
C(3a)–C(3)	1·424 (7)	C(8a)–C(4a)–C(5)	120·3 (5)
C(21)–C(3)	1·368 (7)	N(4)–C(4a)–C(5)	118·3 (4)
N(4)–C(3a)	1·300 (5)	N(4)–C(4a)–C(8a)	121·4 (4)
N(10)–C(3a)	1·376 (6)	C(6)–C(5)–C(4a)	119·7 (5)
C(5)–C(4a)	1·395 (6)	C(7)–C(6)–C(5)	119·8 (5)
C(8a)–C(4a)	1·402 (7)	C(8)–C(7)–C(6)	120·5 (6)
N(4)–C(4a)	1·385 (7)	C(8a)–C(8)–C(7)	120·5 (5)
C(6)–C(5)	1·383 (9)	C(8)–C(8a)–C(4a)	119·2 (4)
C(7)–C(6)	1·403 (8)	C(9)–C(8a)–C(4a)	120·3 (5)
C(8)–C(7)	1·367 (7)	C(9)–C(8a)–C(8)	120·5 (5)
C(8a)–C(8)	1·392 (8)	N(10)–C(9)–C(8a)	113·2 (4)
C(9)–C(8a)	1·456 (6)	O(01)–C(9)–C(8a)	126·3 (5)
N(10)–C(9)	1·375 (8)	O(01)–C(9)–N(10)	120·5 (4)
O(01)–C(9)	1·226 (6)	C(23)–C(21)–C(3)	127·9 (4)
C(23)–C(21)	1·469 (8)	O(22)–C(21)–C(3)	119·2 (5)
O(22)–C(21)	1·352 (5)	O(22)–C(21)–C(23)	112·9 (4)
C(24)–C(23)	1·393 (8)	C(24)–C(23)–C(21)	117·8 (4)
C(28)–C(23)	1·382 (7)	C(28)–C(23)–C(21)	124·2 (5)
C(25)–C(24)	1·372 (10)	C(28)–C(23)–C(24)	118·0 (5)
C(26)–C(25)	1·370 (8)	C(25)–C(24)–C(23)	121·2 (5)
C(27)–C(26)	1·374 (9)	C(26)–C(25)–C(24)	120·1 (6)
C(28)–C(27)	1·380 (9)	C(27)–C(26)–C(25)	119·6 (6)
		C(28)–C(27)–C(26)	120·6 (5)
N(10)–C(1)–C(2)	104·2 (3)	C(27)–C(28)–C(23)	120·5 (5)
C(3)–C(2)–C(1)	104·9 (4)	C(4a)–N(4)–C(3a)	117·6 (4)
C(3a)–C(3)–C(2)	108·6 (4)	C(3a)–N(10)–C(1)	112·5 (4)
C(21)–C(3)–C(2)	130·8 (5)	C(9)–N(10)–C(1)	123·8 (4)
C(21)–C(3)–C(3a)	120·6 (4)	C(9)–N(10)–C(3a)	123·7 (4)

Discussion. Atomic coordinates are given in Table 1.* Table 2 gives the bond lengths and angles of the compound. Fig. 1 shows the molecule. The X-ray analysis has proven the molecule to be the (*Z*) isomer of (II), stabilized by a hydrogen bond between O(22) and N(4) [2·571 (6) Å] as previously postulated (Dunn & Kinnear, 1984). O(22)—H(221) 1·031 (10), N(4)···H(221) 1·698 (10) Å. The phenyl group C(23)···C(28) is close to coplanar with the pyrroloquinazolinone fragment giving the maximum extended π bonding. [The torsion angle C(3)—C(21)—C(23)—C(24) is 171·27 (7)°, angle between the normals to the mean planes of the two benzene rings 7·55 (8)°.]

A number of close contacts exist between O(22)(x, $\frac{1}{2}-y$, $\frac{1}{2}+z$) and atoms C(9) [3·087 (5) Å], N(10 [3·146 (6) Å] and O(01) [3·348 (5) Å] in the parent molecule. A further short contact exists from O(22)-($1-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$) to C(1) [3·289 (7) Å] in the parent molecule.

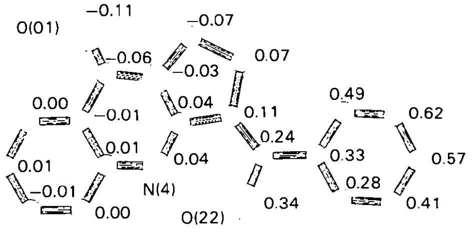


Fig. 1. Molecule viewed perpendicular to the plane C(4a)—C(5)—C(6)—C(7)—C(8)—C(8a). Heights of atoms from this plane are given in Å.

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39830 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of *cyclo*(-α-Aminoisobutyryl-L-phenylalanyl-), $C_{13}H_{16}N_2O_2$

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Abstract. $M_r = 232{\cdot}27$, monoclinic, $P2_1$, $a = 9{\cdot}646\ (4)$, $b = 6{\cdot}133\ (3)$, $c = 10{\cdot}757\ (6)$ Å, $\beta = 101{\cdot}97\ (4)°$, $U = 622{\cdot}5$ Å^3, $Z = 2$, $D_m = 1{\cdot}25$, $D_x = 1{\cdot}24$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}605$ mm^{-1}, $F(000) = 248$, room temperature, $R = 0{\cdot}065$ for 820 significant reflections. The peptide units are planar. The diketopiperazine ring assumes a flat boat conformation. The phenylalanyl ring shows a folded geometry with $\chi^1 = 64{\cdot}6\ (7)$ and $\chi^2 = 96{\cdot}4\ (7)°$.

Introduction. The structure analysis of the title compound [*cyclo*(-Aib-L-Phe-)] was undertaken as a continuation of our investigations on the structures of diketopiperazines (Suguna, Ramakumar, Shamala, Venkataram Prasad and Balaram, 1982) and of peptides containing the α-aminoisobutyric acid (Aib) residue (Prasad & Balaram, 1984). The structure is also of interest since diketopiperazines containing aromatic rings have been extensively studied as models for the interaction of the rings with the peptide bond (Ramani, Sasisekharan & Venkatesan, 1977). *cyclo*(-Aib-L-Phe-) was synthesized as described earlier for related compounds (Nagaraj & Balaram, 1977).

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