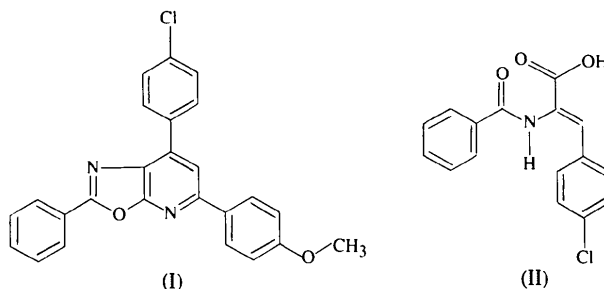


- Klunder, A. J. H., Bos, W. & Zwanenburg, B. (1981). *Tetrahedron Lett.* **22**, 4557–4560.
- Klunder, A. J. H., Zwanenburg, B. & Liu, Z. Y. (1991). *Tetrahedron Lett.* **32**, 3131–3132.
- Lehman, M. S. & Larsen, F. K. (1974). *Acta Cryst.* **A30**, 580–584.
- Liu, Z. Y. & Chu, X. J. (1993). *Tetrahedron Lett.* **34**, 3885–3888.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* Univ. of Göttingen, Germany.
- Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.
- Takano, S., Moriya, M. & Ogasawara, K. (1992). *Tetrahedron Lett.* **33**, 1909–1910.
- Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.
- Zhu, J., Klunder, A. J. H. & Zwanenburg, B. (1994a). *Tetrahedron Lett.* **50**, 10597–10610.
- Zhu, J., Klunder, A. J. H. & Zwanenburg, B. (1994b). *Tetrahedron.* In preparation.

proposed structure in which the two N-containing rings are broken. How this cleavage occurred remains open to conjecture.



The phenyl and chlorophenyl rings are planar. There are no significant deviations from the average bond lengths [1.380 (7) and 1.375 (7) Å]. The bond angles C13—C12—C17 [117.9 (4)°] and C3—C4—C5 [116.7 (4)°] in these rings show slight deviations from the average bond angles of 120.0 (4) and 120.0 (5)°, respectively. These distortions are in keeping with the findings of Domenicano, Murray-Rust & Vaciago (1983). The C—Cl bond length [1.741 (5) Å] is typical of C—Cl bond lengths in structures of the type C(ar)—Cl (Allen *et al.*, 1987). The Cl atom is displaced by 0.086 (7) Å from the least-squares plane calculated for the phenyl ring, the Cl atom and C7 (r.m.s. deviation of all eight atoms is 0.013 Å). The backbone chain of the molecule, C4—C12, is in an extended conformation between C7 and C12, while C4 is *cis* to N10, by rotation about C7—C8 (Table 2).

Hydrogen bonds involving the OH (donor) groups of the carboxylic acid moieties, the carbonyl O atoms (acceptor) of the carboxylic acid moieties and the Cl atoms (acceptor) of the chlorophenyl groups, together with hydrogen bonds between the amino N atoms (donor) and

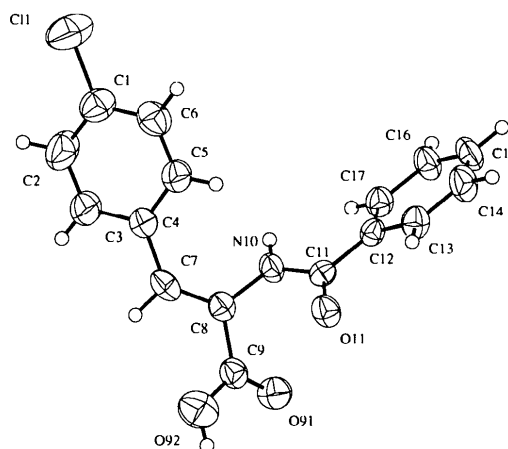


Fig. 1. Minimum overlap view of the molecule showing 50% probability displacement ellipsoids.

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2-Benzamido-3-(*p*-chlorophenyl)propenoic Acid (Antrex)

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Abstract

The phenyl and chlorophenyl rings are planar in the title compound, C₁₆H₁₂ClNO₃. The structure is stabilized by intermolecular hydrogen bonds involving the OH (donor) groups of the carboxylic acid moieties, the carbonyl O atoms (acceptor) of the carboxylic acid moieties and the Cl atoms (acceptor) of the chlorophenyl groups, together with hydrogen bonds between the amino N atoms (donor) and the carbonyl O atoms (acceptor) of the benzamide moieties.

Comment

In an attempt to prepare the compound 7-(4-chlorophenyl)-5-(4-methoxyphenyl)-2-phenyloxazo[5,4-*b*]pyridine, (I), some colourless crystals were obtained (sample supplied by Dr V. Bansal, University of Rajasthan). The crystal structure determination was undertaken to obtain information for use in structure–function studies of 2-phenyloxazo[5,4-*b*]pyridine compounds thought to possess analgesic properties (Clark *et al.*, 1978). The result of this analysis indicates that the compound is 2-benzamido-3-(*p*-chlorophenyl)propenoic acid, (II), which is a cleaved version of the originally

the carbonyl O atoms (acceptor) of the benzamide moieties, stabilize the crystal structure (Table 2). H92 on the carboxy atom O92 occupies two alternative sites, H92A and H92B (50% each), in order to satisfy the hydrogen bonding. Coordinates for H92A and H92B were derived geometrically and their parameters were not allowed to refine.

Experimental

Crystal data

C₁₆H₁₂ClNO₃
M_r = 301.72
 Tetragonal
*I*4₁/*a*
a = 21.184 (5) Å
c = 15.412 (4) Å
V = 6916.3 (29) Å³
Z = 16
D_x = 1.159 Mg m⁻³

Data collection

Enraf-Nonius CAD-4
 diffractometer
 ω -2 θ scans
 Absorption correction:
 none
 3218 measured reflections
 1559 independent reflections
 1329 observed reflections
 [*I* > 2 σ (*I*)]

Refinement

Refinement on *F*²
R[*F*² > 2 σ (*F*²)] = 0.0695
wR(*F*²) = 0.1838
S = 1.128
 1559 reflections
 203 parameters
 $w = 1/[\sigma^2(F_o^2) + (0.1307P)^2 + 9.4711P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.229$
 $\Delta\rho_{\max} = 0.558 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.261 \text{ e } \text{\AA}^{-3}$

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
C11	0.37761 (9)	-0.30240 (7)	0.41604 (12)	0.0911 (8)
C1	0.3589 (3)	-0.2224 (3)	0.4116 (3)	0.061 (2)
C2	0.4025 (2)	-0.1797 (2)	0.3828 (3)	0.082 (2)
C3	0.3866 (2)	-0.1169 (2)	0.3759 (3)	0.068 (2)
C4	0.3277 (2)	-0.0951 (2)	0.3993 (3)	0.0463 (13)
C5	0.2852 (2)	-0.1395 (2)	0.4304 (3)	0.0527 (14)
C6	0.3009 (3)	-0.2020 (3)	0.4382 (3)	0.060 (2)
C7	0.3135 (2)	-0.0274 (2)	0.3929 (3)	0.0467 (13)
C8	0.2582 (2)	0.0019 (2)	0.3825 (3)	0.0401 (12)
C9	0.2547 (2)	0.0720 (2)	0.3785 (3)	0.0433 (13)

O91	0.2173 (2)	0.0985 (2)	0.3291 (2)	0.0566 (10)
O92	0.2935 (2)	0.1032 (2)	0.4307 (3)	0.0809 (13)
N10	0.2021 (2)	-0.0309 (2)	0.3645 (2)	0.0406 (11)
C11	0.1474 (2)	-0.0192 (2)	0.4057 (3)	0.0355 (12)
O11	0.14553 (13)	0.0189 (2)	0.4669 (2)	0.0470 (10)
C12	0.0914 (2)	-0.0546 (2)	0.3792 (3)	0.0349 (12)
C15	-0.0173 (2)	-0.1221 (2)	0.3396 (4)	0.0555 (15)
C13	0.0423 (2)	-0.0606 (2)	0.4385 (3)	0.0469 (13)
C16	0.0306 (2)	-0.1168 (2)	0.2791 (3)	0.0492 (14)
C17	0.0845 (2)	-0.0837 (2)	0.2979 (3)	0.0433 (13)
C14	-0.0114 (2)	-0.0940 (2)	0.4180 (4)	0.0563 (15)

Table 2. Selected geometric parameters (Å, °)

C11—C1	1.741 (5)	C9—O91	1.234 (6)
C1—C2	1.367 (7)	C9—O92	1.327 (6)
C1—C6	1.365 (8)	N10—C11	1.344 (6)
C2—C3	1.376 (6)	C11—O11	1.242 (5)
C3—C4	1.379 (6)	C11—C12	1.460 (6)
C4—C5	1.386 (7)	C12—C13	1.391 (6)
C4—C7	1.468 (7)	C12—C17	1.405 (6)
C5—C6	1.371 (7)	C15—C14	1.352 (7)
C7—C8	1.336 (7)	C15—C16	1.383 (7)
C8—N10	1.404 (6)	C13—C14	1.377 (7)
C8—C9	1.489 (7)	C16—C17	1.371 (6)
C2—C1—C6	119.7 (5)	O91—C9—C8	120.7 (4)
C2—C1—C11	120.2 (4)	O92—C9—C8	116.1 (4)
C6—C1—C11	120.1 (4)	C11—N10—C8	123.1 (4)
C1—C2—C3	119.9 (3)	O11—C11—N10	120.3 (4)
C2—C3—C4	121.7 (3)	O11—C11—C12	121.3 (4)
C3—C4—C5	116.7 (4)	N10—C11—C12	118.3 (4)
C3—C4—C7	119.6 (4)	C13—C12—C17	117.9 (4)
C5—C4—C7	123.6 (4)	C13—C12—C11	118.1 (4)
C6—C5—C4	121.9 (5)	C17—C12—C11	124.0 (4)
C1—C6—C5	119.9 (5)	C14—C15—C16	119.9 (4)
C8—C7—C4	129.9 (4)	C14—C13—C12	121.0 (4)
C7—C8—N10	122.4 (4)	C17—C16—C15	120.7 (4)
C7—C8—C9	120.9 (4)	C16—C17—C12	120.0 (4)
N10—C8—C9	116.3 (4)	C15—C14—C13	120.5 (5)
O91—C9—O92	123.1 (4)	C7—C8—C9—O92	-37.6 (6)
C6—C1—C2—C3	-3.8 (6)	N10—C8—C9—O92	149.8 (4)
C2—C3—C4—C7	178.4 (3)	C7—C8—N10—C11	134.2 (5)
C3—C4—C5—C6	0.5 (7)	C9—C8—N10—C11	-53.4 (6)
C7—C4—C5—C6	-177.6 (5)	C8—N10—C11—O11	-5.2 (6)
C11—C1—C6—C5	-176.5 (4)	C8—N10—C11—C12	177.7 (4)
C3—C4—C7—C8	154.4 (5)	O11—C11—C12—C13	-20.1 (6)
C5—C4—C7—C8	-27.5 (8)	N10—C11—C12—C13	157.0 (4)
C4—C7—C8—N10	-8.4 (8)	O11—C11—C12—C17	160.9 (4)
C4—C7—C8—C9	179.4 (4)	N10—C11—C12—C17	-22.0 (6)
C7—C8—C9—O91	142.7 (5)	C11—C12—C17—C16	179.0 (4)
N10—C8—C9—O91	-29.9 (6)		

Table 3. Hydrogen-bonding geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O92—H92A...C11 ⁱ	0.82	3.327 (2)	3.577 (4)	101.1 (3)
O92—H92B...O91 ⁱⁱ	0.86	2.232 (3)	2.971 (5)	143.6 (3)
N10—H10...O11 ⁱⁱⁱ	0.86	2.007 (5)	2.821 (5)	157.3 (4)

Symmetry codes: (i) $x, \frac{1}{2} + y, 1 - z$; (ii) $\frac{1}{4} + y, \frac{1}{4} - x, \frac{1}{4} + z$; (iii) $\frac{1}{4} - y, x - \frac{1}{4}, z - \frac{1}{4}$.

H atoms were refined in riding mode with *U*₁₁ variable, except for H92A and H92B which are modelled with (unrefined) half-occupied alternative sites on O92 consistent with hydrogen bonding. The quality of the refinement was limited by the relatively weak diffracting power of the crystals, intensity data being measurable only to ca 50° θ .

The structure was solved by direct methods using the program *SHELXS86* (Sheldrick, 1985). Refinement was by full-matrix least squares using *SHELXL93* (Sheldrick, 1993), with which geometrical calculations were also performed. Molecular illustrations were drawn using *SNOOPI* (Karaulov, 1992). Calculations were carried out on a VAX 11/750 computer and Löfgren PC486.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1184). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
- Clark, R. L., Arsenio, A., Pessolano, A., Witzel, B., Lanza, T. & Shen, T. Y. (1978). *J. Med. Chem.* **21**, 1158.
- Domenicano, A., Murray-Rust, P. & Vaciago, A. (1983). *Acta Cryst.* **B39**, 457–468.
- Karaulov, S. (1992). *SNOOPI. Molecular Plotting Program*. Univ. of Wales, Cardiff, Wales.
- Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

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(S)-[1-(Benzyloxycarbonylamino)ethyl]-phosphonic Acid

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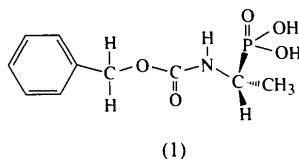
Abstract

The title compound, C₁₀H₁₄NO₅P, is one of a series of novel carbonic anhydrase inhibitors [Ösabay & Csiba (1993). *Eur. J. Med. Chem.* **28**, 355–361]. In an initial approach to structure–activity relationship studies, the molecular structure of the title compound was determined by X-ray crystallography. The molecule is fully extended with a *trans* urethane moiety. The phosphonate group emerges from the plane of the backbone with a 92.3° torsion angle. The orientation of phosphonate groups facilitates the formation of two intermolecular hydrogen bridges (1.812 and 2.253 Å) between adjacent phosphonate moieties.

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Comment

A series of phosphonic analogs of amino acids and peptides showed significant inhibitory potency for the esterase activity of the human carbonic anhydrase (HCA). The title compound, (S)-[1-(benzyloxycarbonylamino)ethyl]phosphonic acid, (1), possesses the strongest inhibitory activity toward both HCA isoenzymes I and II. The structure determination of (1) was undertaken within the context of investigations on a hypothetical inhibitor–enzyme complex by computer directed analysis (Ösabay & Csiba, 1993).



Intramolecular bond distances and angles, listed in Table 2, do not deviate significantly from the expected values (*Chemistry Data Book*, 1982). The molecule has a fully extended structure with *trans* backbone torsion angles: C(8)—O(1)—C(7)—C(6) –166.6, C(7)—O(1)—C(8)—N(1) –179.1 and C(9)—N(1)—C(8)—O(1) –179.2°. The aromatic ring is planar within experimental error and it is in a *gauche*[–] position relative to the CH₂—O bond; the torsion angle C(1)—C(6)—C(7)—O(1) is –67.8°. The chain O(5)—P(1)—C(9)—N(1) is close to planar with a torsion angle of 167.7°, and the torsion angle C(8)—N(1)—C(9)—P(1) of –92.3° indicates that the phosphonate group emerges from the plane of the backbone. The P atom has an approximate tetrahedral environment of one C and three O atoms. The P(1)—C(9) distance has a value of 1.834 (5) Å, which is close to that of 1.82 Å in aminomethylphosphonic acid (Darriet, Darriet, Cassaigne & Neuzil, 1975). There are two types of P—O bonds, which are affected by their participation in intermolecular hydrogen-bridge formation. The two HO—P bonds have lengths of 1.552 (4) [P(1)—O(5)] and 1.529 (4) Å [P(1)—O(4)] (P—OH bonds are found in the range 1.56–1.57 Å in the literature), while the P=O bond is

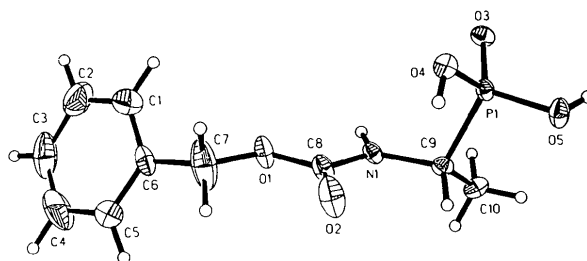


Fig. 1. ORTEP (Johnson, 1965) view of the title compound. The atoms are drawn with 30% probability ellipsoids.