Table 3. *Torsion angles* (°) *in the title compound* (I) *and ramiprilat* (II)

	(I)	(II)
C1–C2–N2–C3	−43·1	−81·4
C2–N2–C3–C4	4·6	−175·1
N2–C3–C4–N1	35·7	130·8
C3–C4–N1–C11	149·7	−56·6
C4–N1–C11–C12	101·6	157·6
N1–C11–C12–C13	−65·4	66·6
C11–C12–C13–C17	178·6	173·4

condensation. There seems to be no opportunity for easy condensation to occur as apparently happens in ramipril. Because the differences in the structural formulae are quite small, the influence of the only group which is different between the molecules is promoting the dramatic changes. Ramipril has a betaine structure: the ammonium group and the carboxylate group are then 'predestined' to approach each other and split off a water molecule. The situation is different in ramiprilat (II): there is now an additional carboxy group quite near to the amino group. It is not so easy for the carboxy group at the pyrrolidine ring to react with the amino group, because it is far away and rejected by the other carboxy group which is not esterified in ramiprilat (II). Nevertheless this reaction is possible by refluxing in toluene and removing the produced water out of equilibrium.

The crystal structure of (I) is represented in Fig. 2, which is a projection of the structure along the *a* axis. The screw axes parallel to the three crystallographic directions can be recognized quite well. The intermolecular distances between non-hydrogen atoms are greater than 3·3 Å.

References

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99, 149. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

PAULUS, E. F., HENNING, R. & URBACH, H. (1987). *Acta Cryst.* C**43**, 941–945.

SHELDRICK, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen.

TEETZ, V., GEIGER, R., HENNING, R. & URBACH, H. (1984). *Arzneim. Forsch. Drug Res.* **34**(11), 1399–1401.

Acta Cryst. (1987). C**43**, 941–945

Structure of the Angiotensin-Converting Enzyme Inhibitor Ramiprilat (HOE 498 Diacid)

BY E. F. PAULUS, R. HENNING AND H. URBACH

Hoechst AG, D-6230 *Frankfurt am Main* 80, *Federal Republic of Germany*

(*Received* 3 *November* 1985; *accepted* 4 *December* 1986)

Abstract. HOE 498 diacid, 2-{*N*-[(*S*)-1-carboxy-3-phenylpropyl]-L-alanyl}-(1*S*,3*S*,5*S*)-2-azabicyclo-[3.3.0]octane-3-carboxylic acid, $C_{21}H_{28}N_2O_5$, M_r = 388·47, crystallizes with 4·7 molecules of methanol in the orthorhombic space group $P2_12_12_1$ (M_r of complex = 539·07), a = 10·529 (1), b = 12·147 (2), c = 22·240 (3) Å, V = 2844·4 Å^3, Z = 4, D_x = 1·28 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 0·079 mm^{-1}, T = 295 K, R = 0·112, wR = 0·042, 2794 observed reflections [$F^2 > \sigma(F^2)$]. It was possible to determine the given absolute configuration because L-alanine was one of the components of the synthesis [Teetz, Geiger, Henning & Urbach (1984). *Arzneim. Forsch. Drug Res.* **34**(11), 1399–1401]. The pyrrolidine and the cyclopentane rings of the bicyclooctane ring system have an envelope and a twist conformation respectively. The consequence of the *endo* position of the carboxyl group at C1 is that the methylene group C6 is forced to assume an *exo* conformation. To a large extent, the crystal structure is influenced by the non-stoichiometric content of methanol molecules in channels. Proof of the mobility of these molecules is given by the extremely large temperature coefficients (as large as 1·38 Å^2) and the rapid crystal decomposition in a methanol-free atmosphere.

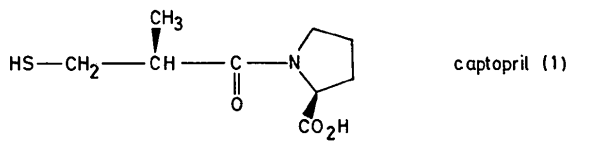
Introduction. The inhibition of angiotensin-converting enzyme (ACE, kininase II, EC 3.4.15.1) represents a new therapeutical principle for the treatment of hypertension and cardiac insufficiency (Stumpe, Overlack & Kolloch, 1984).

As a peptidyl dipeptide carboxyhydrolase ACE hydrolyses the *C*-terminal dipeptide from the angiotensin 1 decapeptide to yield the potent vasopressor octapeptide angiotensin II (Petrillo & Ondetti, 1982). It also destroys the blood-pressure-lowering effect of the hypotensive nonapeptide bradykinin by splitting off the *C*-terminal dipeptide to a biologically inactive heptapeptide (Petrillo & Ondetti, 1982). Inhibitors of ACE consequently will have an antihypertensive effect. The

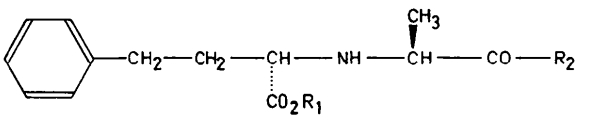
0108-2701/87/050941-05$01.50

development of such inhibitors led to the proline derivatives captopril (1) (Cushman, Cheung, Sabo & Ondetti, 1977), enalapril (2) (Patchett *et al.*, 1980) and numerous other ACE inhibitors (Wyvratt & Patchett, 1985). Clinical studies revealed the usefulness of such compounds for the control of high blood pressure and congestive heart failure.

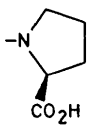
It is known that hydrophobic interactions play an important role in the binding of substrates or inhibitors to the active site of an enzyme. In pursuit of this general knowledge we synthesized the more lipophilic and more sterically pretentious ACE inhibitor ramipril (4) (Teetz, Geiger, Henning & Urbach, 1984). It can be shown that ramiprilate (5), the active principle of (4) (Becker, Schölkens, Metzger & Schulze, 1984), is a reversible and competitive inhibitor (Bünning, 1984). It interacts with ACE by rapidly forming an initial enzyme–inhibitor complex which then undertakes a slow isomerization reaction. The overall inhibition constant is similar to those obtained for (1) and enalaprilat (3), the active principle of (2).



captopril (1)

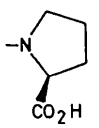


R_1= $-C_2H_5$, R_2 =



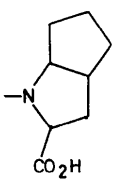
enalapril (2)

R_1 = $-H$, R_2 =



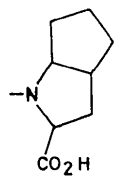
enalaprilat (3)

R_1 = $-C_2H_5$, R_2 =



ramipril (4)

R_1 = $-H$, R_2 =



ramiprilat (5)

Therefore we have analysed the crystal structure of (5) to compare it with that of (3) (Wyvratt *et al.*, 1984). We wanted to see whether there are differences in conformation of the molecules, which could possibly give some hints as to the differences in the overall inhibition constants.

Because L-alanine was used for the synthesis of (5), the relative configuration will reveal the absolute one at the same time.

Experimental. The sample was recrystallized from methanol. The crystals are extremely unstable in air and decompose, losing methanol. A crystal of dimensions $0{\cdot}5 \times 0{\cdot}2 \times 0{\cdot}1$ mm was therefore sealed into a Mark tube together with a drop of methanol. 25 reflections with $\theta > 9{\cdot}5°$ were used for the cell refinement. Nicolet $R3$ computer-controlled diffractometer, $2\theta/\theta$ scan, $\theta_{max} = 28°$, $3°$ min^{-1}; no correction for absorption or extinction; one standard reflection (106), intensity variation 1·5%. 3875 unique reflections measured, of which 2794 were considered observed $[F^2 > \sigma(F^2)]$. h 0→13, k 0→16, l 0→29. Solution of the phase problem with the program *SHELXTL* (Sheldrick, 1983) was quite lengthy. Together with the three origin-fixing phases ten other starting reflections were necessary to produce 267 phase sets, from which the one with the best figures of merit could be used to calculate an E map. The interpretation was quite ambiguous. After a few further difference Fourier syntheses we were able to find all the atoms of the molecule together with a few small peaks which had to be interpreted as methanol atoms. After a few cycles of least squares the hydrogen atoms could not be found in a difference Fourier synthesis and were therefore given calculated atomic coordinates assuming C—H distances of 0·96 Å. The hydrogens at the O atoms were omitted.

Refinement of the coordinates of the methanol atoms was quite troublesome. Six extra maxima could be seen in the difference Fourier synthesis, far from the molecule; four of them could be allocated to two methanol molecules, whereas the other two seemed to belong to two different methanol molecules. No convergence was obtained by refining all the non-hydrogen atoms anisotropically. We did, however, obtain a convergence when the multiplicity of the methanol atoms was also refined. Table 1 gives the result.

Tests showed that none of these atoms needs to be substituted by a much heavier atom. The distinction between carbon and oxygen is a kind of formality. The multiplicity tells us that within the ellipsoid defined by the temperature coefficients there is more than one atom, in the extreme case one methanol molecule. Because the structure is non-centrosymmetric, it is not advisable to refine the structure without these methanol atoms; the phases would be wrong because essential parts of the structure would be missing.

The R and wR values were 0·112 and 0·042 respectively. Refinement was based on F. The weighting scheme was $w = 1/\sigma^2(F)$, where σ was calculated according to the counting statistics of the intensity measurements.* Because there are many weak reflections and reflections with intensities as low as $\sigma(F^2)$ were taken, the difference between R and wR is quite large. The ten largest peaks in the final difference electron density synthesis were between 0·25 and 0·39 e Å^{-3}. $(\Delta/\sigma)_{max}<0·1$. All the calculations and drawings were performed with a Nova 3/12 computer and *SHELXTL* (Sheldrick, 1983); the scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

* Lists of structure factors, anisotropic temperature coefficients, hydrogen-atom coordinates, further bond lengths and angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43622 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Results of refinement of the multiplicity of atoms of methanol molecules*

	Multiplicity	Temperature coefficient (Å²)	Shortest distance from host atom (Å)
C22	1·10	8·2	2·096 (H2)
C23	1·12	7·0	2·679 (O2)
O8	2·07	109·5	3·297 (H03)
O6	1·66	35·8	3·109 (H2)
O7	1·56	16·7	3·244 (H21)
C24	1·93	39·6	2·973 (O1)

Table 2. *Final atomic coordinates and equivalent isotropic temperature factors*

The atomic coordinates are given in units of the lattice parameters, the isotropic temperature coefficients were calculated from the anisotropic ones after the last least-squares cycle.

$U = \frac{1}{3}$ (trace $\tilde{\mathbf{U}}$).

	x	y	z	U(Å²)
O1	0·3978 (3)	0·4276 (3)	0·0453 (1)	0·067
O2	0·6783 (3)	0·2186 (2)	0·1970 (1)	0·062
O3	0·5059 (3)	0·2713 (3)	0·2472 (1)	0·065
O4	0·1870 (3)	0·2760 (3)	−0·0429 (1)	0·072
O5	0·3401 (3)	0·2666 (3)	−0·1104 (1)	0·065
O6	0·4358 (4)	0·6538 (3)	0·1547 (2)	0·104
O7	0·6429 (3)	0·0045 (3)	0·2208 (1)	0·089
O8	0·5283 (17)	0·8051 (7)	−0·0400 (6)	1·380
N1	0·3596 (3)	0·2460 (3)	0·0505 (1)	0·043
N2	0·4402 (3)	0·4267 (3)	0·1697 (1)	0·049
C1	0·3981 (4)	0·2299 (4)	−0·0104 (2)	0·051
C2	0·4310 (5)	0·1057 (4)	−0·0145 (2)	0·067
C3	0·3564 (5)	0·0533 (4)	0·0368 (2)	0·076
C4	0·3305 (5)	0·1451 (4)	0·0821 (2)	0·061
C5	0·1871 (5)	0·1322 (5)	0·0997 (2)	0·087
C6	0·1478 (6)	0·0196 (5)	0·0785 (2)	0·108
C7	0·2208 (5)	0·0105 (5)	0·0192 (2)	0·105
C8	0·2966 (4)	0·2609 (4)	−0·0562 (2)	0·052
C9	0·3662 (4)	0·3464 (4)	0·0743 (2)	0·051
C10	0·3350 (4)	0·3604 (4)	0·1409 (2)	0·048
C11	0·5693 (4)	0·3753 (3)	0·1613 (2)	0·044
C12	0·6757 (4)	0·4629 (4)	0·1680 (2)	0·060
C13	0·6880 (4)	0·5094 (4)	0·2294 (2)	0·071
C14	0·2091 (4)	0·4229 (5)	0·1493 (2)	0·076
C15	0·5839 (4)	0·2812 (3)	0·2059 (2)	0·052
C16	0·8038 (5)	0·5838 (4)	0·2327 (2)	0·068
C17	0·9208 (5)	0·5429 (5)	0·2504 (3)	0·095
C18	1·0242 (5)	0·6128 (5)	0·2535 (3)	0·099
C19	1·0166 (6)	0·7184 (5)	0·2375 (3)	0·120
C20	0·9021 (6)	0·7611 (5)	0·2202 (3)	0·117
C21	0·7970 (5)	0·6917 (4)	0·2181 (3)	0·092
C22	0·3865 (11)	0·7213 (7)	0·1150 (4)	0·453
C23	0·6258 (6)	−0·0376 (5)	0·1597 (2)	0·212
C24	0·7953 (10)	0·8857 (6)	0·0263 (3)	0·502

Discussion. Table 2 lists the final atomic coordinates and Table 3 the important bond lengths and angles. The most characteristic feature of the crystal structure is the presence of the solvent molecules of methanol. Fig. 1 shows that the guest molecules are situated in large channels which are left by the host molecules. The mobility of these methanol molecules is largest in the direction of the channels, as can be seen from the coefficients of the anisotropic temperature factors which are greatest in the **a** direction. From Table 1 one would usually think that at least O8, C22 and C24 would have to be rejected. Leaving only O8 out of the parameter refinement raises the R and wR values from 0·112 and 0·042 to 0·132 and 0·198 respectively. We do not want to claim that the coordinates and temperature coefficients of these atoms have any reality, but we want to point out that we have found a model which converges in the least-squares procedure and which also has some plausibility. The methanol molecules are mostly spread over the channels; the

Table 3. *Bond lengths* (Å) *and bonding angles* (°) *with e.s.d.'s in parentheses*

O1–C9	1·225 (5)	C3–C7	1·569 (8)
O2–C15	1·267 (5)	C4–C5	1·568 (8)
O3–C15	1·238 (5)	C5–C6	1·505 (8)
O4–C8	1·205 (5)	C6–C7	1·530 (7)
O5–C8	1·290 (5)	C9–C10	1·526 (6)
O6–C22	1·311 (10)	C10–C14	1·539 (6)
O7–C23	1·464 (6)	C11–C12	1·552 (6)
N1–C1	1·427 (5)	C11–C15	1·521 (6)
N1–C4	1·445 (6)	C12–C13	1·484 (6)
N1–C9	1·331 (6)	C13–C16	1·519 (7)
N2–C10	1·511 (5)	C16–C17	1·385 (7)
N2–C11	1·508 (5)	C16–C21	1·353 (7)
C1–C2	1·550 (7)	C17–C18	1·383 (7)
C1–C8	1·525 (6)	C18–C19	1·333 (8)
C2–C3	1·523 (7)	C19–C20	1·368 (9)
C3–C4	1·528 (7)	C20–C21	1·391 (8)
O1–C9–N1	122·8 (4)	C2–C3–C4	106·3 (4)
O1–C9–C10	118·7 (4)	C2–C3–C7	114·9 (4)
O2–C15–O3	125·3 (4)	C3–C4–C5	105·2 (4)
O2–C15–C11	115·4 (3)	C3–C7–C6	102·7 (4)
O3–C15–C11	119·3 (4)	C4–N1–C9	126·5 (3)
O4–C8–O5	124·2 (4)	C4–C3–C7	104·1 (4)
O4–C8–C1	122·9 (4)	C4–C5–C6	106·1 (4)
O5–C8–C1	112·9 (3)	C5–C6–C7	101·3 (4)
N1–C1–C2	104·7 (4)	C9–C10–C14	111·0 (4)
N1–C1–C8	113·7 (3)	C10–N2–C11	112·8 (3)
N1–C4–C3	105·1 (4)	C11–C12–C13	114·3 (3)
N1–C4–C5	114·2 (4)	C12–C11–C15	112·4 (3)
N1–C9–C10	118·5 (4)	C12–C13–C16	109·9 (4)
N2–C10–C9	108·2 (3)	C13–C16–C17	121·0 (4)
N2–C10–C14	108·5 (4)	C13–C16–C21	121·5 (4)
N2–C11–C12	110·8 (3)	C16–C17–C18	119·7 (5)
N2–C11–C15	108·8 (3)	C16–C21–C20	122·5 (5)
C1–N1–C4	113·8 (4)	C17–C16–C21	117·5 (5)
C1–N1–C9	119·2 (4)	C17–C18–C19	122·0 (5)
C1–C2–C3	104·3 (4)	C18–C19–C20	119·5 (6)
C2–C1–C8	111·0 (4)	C19–C20–C21	118·7 (5)

crystal structure collapses when the additional hydrogen bonds are missing. The host molecules themselves put up only one relatively quite short intermolecular hydrogen bond between O5 and O2 (2·578 Å), nevertheless there are a few more acceptors and donors for possible intermolecular hydrogen bonds.

The overall geometry of the host molecule can be seen from Fig. 2. The configuration is all-(*S*), the connection of the cyclopentane and pyrrolidine rings is *cis*. The pyrrolidine ring has an envelope and the cyclopentane ring a twist conformation, which is favoured by the *endo* carboxyl group (the H atoms of the carboxyl groups could not be determined). The conformation of the chain between the phenyl ring and the bicyclic ring system is not fully stretched.

Figs. 3 and 4 show least-squares fits of ramiprilat with captopril (Fujinaga & James, 1980) and enalaprilat (Wyvratt *et al.*, 1984) using all the appropriate atoms. It can be seen that we have always the same configuration and very similar conformations. Especially interesting is the fit of ramiprilat and enalaprilat. Ramiprilat has only one cyclopentane ring more, which makes the molecule more lipophilic and at the same time does not change the shape of the molecule. Moreover, it seems that this small change is sterically advantageous for the active site of the enzyme, because the drug is so effective.

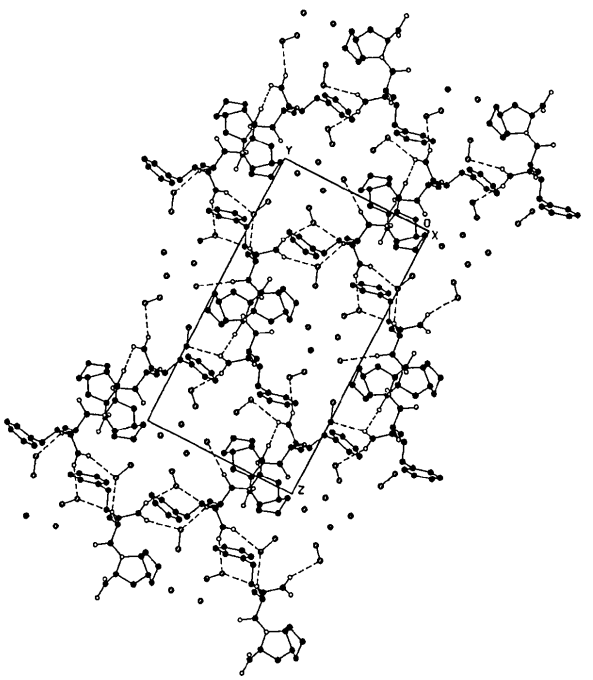


Fig. 1. The crystal structure, projection along the *a* axis, showing the channels for the methanol molecules. For interpretational reasons the O and N atoms are characterized by open circles and the atoms of methanol are shaded.

Additional comparisons of the X-ray structures of different ACE inhibitors and derivatives like HOE 498-dioxopiperazine (Teetz, Geiger, Henning & Urbach, 1984; Paulus, Geiger, Henning, Teetz & Urbach, 1987), YS980 and SA446 (In, Shibata, Doi, Ishida, Inome, Sasaki & Morimoto, 1986) and octahydropyridazo[1,2-*a*]pyridazinediones (Gilbert & Thomas,

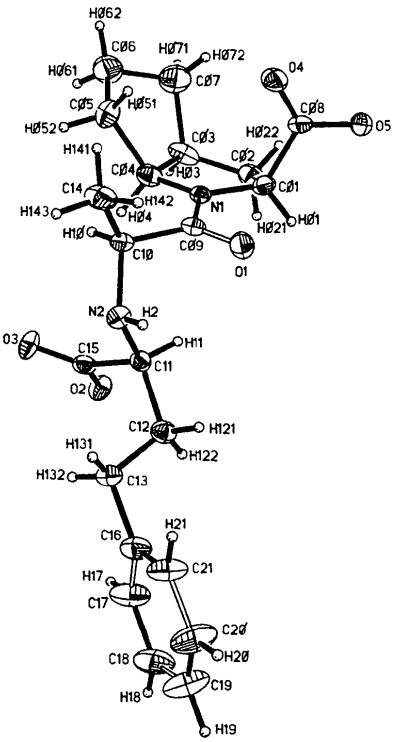


Fig. 2. Molecular structure of HOE 498 diacid, showing 20% probability thermal ellipsoids for the non-hydrogen atoms and the atom-numbering scheme.

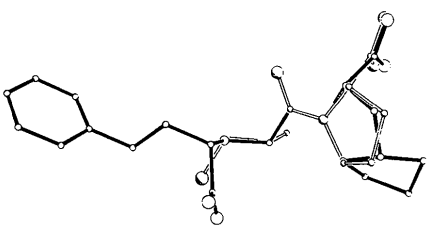


Fig. 3. Least-squares fit of ramiprilat and captopril.

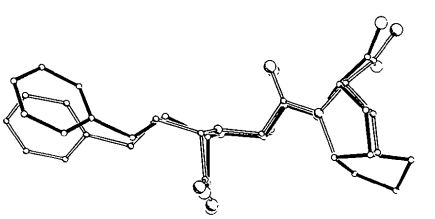


Fig. 4. Least-squares fit of ramiprilat and enalaprilat.

1985) could lead to the design of new biologically active drugs and should help to provide more insight into the nature of the enzyme active site.

References

BECKER, R. H. A., SCHÖLKENS, B. A., METZGER, M. & SCHULZE, K. J. (1984). *Arzneim. Forsch. Drug Res.* **34**(11), 1411–1416.

BÜNNING, P. (1984). *Arzneim. Forsch. Drug. Res.* **34**(11), 1406–1410.

CUSHMAN, D. W., CHEUNG, H. S., SABO, E. F. & ONDETTI, M. A. (1977). *Biochemistry*, **16**, 5484–5491.

FUJINAGA, M. & JAMES, M. N. G. (1980). *Acta Cryst.* B**36**, 3196–3199.

GILBERT, P. J. & THOMAS, W. A. (1985). *J. Chem. Soc. Perkin Trans.* 2, pp. 1077–1082.

IN, Y., SHIBATA, M., DOI, M., ISHIDA, T., INONE, M., SASAKI, Y. & MORIMOTO, S. (1986). *J. Chem. Soc. Chem. Commun.* pp. 473–474.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

PATCHETT, A. A., HARRIS, E., TRISTRAM, E. W., WYVRATT, M. J., WU, M. T., TAUB, D., PETERSOP, E. R., IKELER, T. J., TEN BROEKE, J., PAYNE, L. G., ONDEYKA, D. L., THORSETT, E. D., GREENLEE, W. J., LOHR, N. S., HOFFFSOMER, R. D., JOSHUA, H., RUYLE, W. V., ROTHROCK, J. W., ASTER, S. D., MAYCOCK, A. L., ROBINSON, F. M., HIRSCHMANN, R., SWEET, C. S., ULM, E. H., GROSS, D. M., VASSIL, T. C. & STONE, C. A. (1980). *Nature (London)*, **288**, 280–283.

PAULUS, E. F., GEIGER, R., HENNING, R., TEETZ, V. & URBACH, H. (1987). *Acta Cryst.* C**43**, 938–941.

PETRILLO, E. W. & ONDETTI, M. A. (1982). *Med. Res. Rev.* **2**, 1–41.

SHELDRICK, G. M. (1983). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen.

STUMPE, K. O., OVERLACK, A. & KOLLOCH, R. (1984). *Dtsch. Med. Wochenschr.* **109**, 1295–1299.

TEETZ, V., GEIGER, R., HENNING, R. & URBACH, H. (1984). *Arzneim. Forsch Drug Res.* **34**(11), 1399–1401.

WYVRATT, M. J. & PATCHETT, A. A. (1985). *Med. Res. Rev.* **5**, 483–531.

WYVRATT, M. J., TRISTRAM, E. T., IKELER, T. J., LOHR, N. S., JOSHUA, H., SPRINGER, J. P., ARISON, B. H. & PATCHETT, A. A. (1984). *J. Org. Chem.* **49**, 2816–2819.

Acta Cryst. (1987). C**43**, 945–947

Structure de l'Acide L-Amino-2 Uréido-5 Pentanoïque (L-Citrulline)

PAR P. TOFFOLI, P. KHODADAD ET N. RODIER

Laboratoire de Chimie Minérale, Faculté des Sciences Pharmaceutiques et Biologiques, rue J.-B. Clément, 92296 Châtenay-Malabry CEDEX, France

ET J. ASTOIN

Centre de recherche des Laboratoires Biocodex, 22, rue Jean Bouton, 75012 Paris, France

(*Reçu le 9 juin* 1986, *accepté le 8 décembre* 1986)

Abstract. $C_6H_{13}N_3O_3$, $M_r = 175{\cdot}2$, monoclinic, $P2_1$, $a = 9{\cdot}162$ (5), $b = 5{\cdot}143$ (3), $c = 8{\cdot}969$ (3) Å, $\beta = 95{\cdot}81$ (2)°, $V = 420$ Å^3, $Z = 2$, $D_x = 1{\cdot}384$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}10$ mm^{-1}, $F(000) = 188$, $T = 294$ (1) K, $R = 0{\cdot}029$ for 958 independent reflections [$I > 2\sigma(I)$]. The L-citrulline molecules are zwitterionic. Their geometry is closely related to that of the same molecules in L-citrulline dihydrate. They are linked together by N—H···O hydrogen bonds.

Introduction. La détermination de la structure cristalline de la L-citrulline anhydre s'inscrit dans le cadre de l'étude des combinaisons ou des associations moléculaires formées par cet aminoacide avec les polyacides organiques. Elle fait suite à l'étude de la L-citrulline dihydrate (Toffoli, Rodier & Astoin, 1986). Le produit a été obtenu par évaporation de la solution aqueuse à $T = 338$ K selon la technique indiquée par Ajinomoto Co. (1981).

Partie expérimentale. Cristal en forme de plaquette: 0,05 × 0,15 × 0,40 mm. Dimensions de la maille déterminées sur monocristal avec 25 réflexions telles que $6{,}23 \le \theta \le 16{,}02°$. Diffractomètre Enraf–Nonius CAD-4. $0{,}049 \le (\sin\theta)/\lambda \le 0{,}661$ Å^{-1}. $-12 \le h \le 12$; $0 \le k \le 6$; $0 \le l \le 11$. Réflexions de contrôle: $\bar{3}10$, $\bar{1}\bar{1}3$ et $1\bar{1}3$. $\sigma(I)/I$ moyen (contrôle): 5,1 $\times 10^{-3}$. 1119 réflexions indépendantes mesurées, 161 inobservées [$I \le 2\sigma(I)$]. Méthodes directes programme *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Affinement sur F, programme à matrice entière. Facteurs de diffusion des *International Tables for X-ray Crystallography* (1974) corrigés de f' et de f''. Paramètres affinés: x, y, z de tous les atomes, β_{ij} de C, N, O et B de H. $R = 0{,}029$, $wR = 0{,}037$, $w = 1/\sigma^2(F)$. $S = 1{,}38$. $(\Delta/\sigma)_{max} = 0{,}0$. $|\Delta\rho|_{max} = 0{,}16$ (3) e Å^{-3}. Coefficient d'extinction secondaire isotrope: $g = 2{,}1$ (9) $\times 10^{-6}$. Programmes de calcul du système *SDP* (B. A. Frenz & Associates Inc., 1982)

0108-2701/87/050945-03$01.50