Related literature. The crystals of title compound are isostructural with those of N^1-methyl-N^1-phenyl-N^2-(*p*-tolyl)benzamidine (Oszczapowicz, Tykarska, Jaskólski & Kosturkiewicz, 1986). The configuration around C=N^2 is the same as in N^2-(*p*-methoxyphenyl)-N^1,N^1-pentamethylenebenzamidine (Tykarska, Jaskólski & Kosturkiewicz, 1986) and N^1,N^2-diphenylbenzamidine (Alcock, Barker & Kilner, 1988), but opposite to that in the N^2-*p*-nitrophenylbenzamidine (Surma, Jaskólski, Kosturkiewicz & Oszczapowicz, 1988). The rules governing the configuration of amidines are discussed by Ciszak, Gdaniec, Jaskólski, Kosturkiewicz, Owsiański & Tykarska (1989).

The authors acknowledge the support of program RP.II.10.6.1.5. (MEN).

References

ALCOCK, N. W., BARKER, J. & KILNER, M. (1988). *Acta Cryst.* C**44**, 712–715.

CISZAK, E., GDANIEC, M., JASKÓLSKI, M., KOSTURKIEWICZ, Z., OWSIAŃSKI, J. & TYKARSKA, E., (1989). *Acta Cryst.* C**45**, 433–438.

JASKÓLSKI, M. (1982). *Fourth Symposium on Organic Crystal Chemistry*, Poznań, September 1982, edited by Z. KAŁUSKI, pp. 70–71. Adam Mickiewicz Univ., Poland.

LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* A**30**, 580–584.

OSZCZAPOWICZ, J., RACZYŃSKA, E. & ORLIŃSKI, R. (1981). *Pol. J. Chem.* **55**, 2031–2037.

OSZCZAPOWICZ, J., TYKARSKA, E., JASKÓLSKI, M. & KOSTURKIEWICZ, Z. (1986). *Acta Cryst.* C**42**, 1816–1818.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

SURMA, K., JASKÓLSKI, M., KOSTURKIEWICZ, Z. & OSZCZAPOWICZ, J. (1988). *Acta Cryst.* C**44**, 1031–1033.

TYKARSKA, E., JASKÓLSKI, M. & KOSTURKIEWICZ, Z. (1986). *Acta Cryst.* C**42**, 740–743.

Acta Cryst. (1989). C**45**, 1649–1651

Structure of *N*-(*tert*-Butoxycarbonyl)kainic Acid 2-Diphenylmethyl Ester*

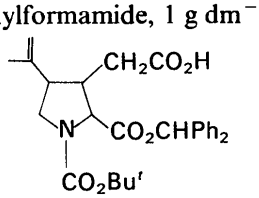
BY D. PAPAIOANNOU, E. SIVVAS, V. NASTOPOULOS, M. SEMERTZIDIS AND S. VOLIOTIS

Department of Chemistry, University of Patras, GR-26110 Patras, Greece

(*Received* 15 *December* 1988; *accepted* 4 *April* 1989)

Abstract. $C_{28}H_{33}NO_6$, $M_r = 479{\cdot}54$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}694\ (2)$, $b = 15{\cdot}397\ (3)$, $c = 20{\cdot}120\ (4)$ Å, $V = 2693{\cdot}3\ (9)$ Å^3, $Z = 4$, $D_x = 1{\cdot}183$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}77$ cm^{-1}, $F(000) = 1024$, room temperature, $R = 0{\cdot}044$ for 1514 unique observed reflections. The atoms N8, C9, C11 and C12 of the proline ring (O_2C—C12—N8—C9—C10—C11) lie in a plane to within ±0·07 Å, while C10 is displaced out of the plane by 0·62 Å. The dihedral angle between the plane of the three atoms C9, C10 and C11 and the plane of the above-mentioned four atoms of the proline ring is 140 (1)°. The structure is stabilized in the **a** direction by means of intermolecular hydrogen bonds [OH18···O7 = 2·05 (7) Å]. Bond lengths and angles are normal.

Experimental. Recrystallization from ethyl acetate–petroleum ether 40–60°, m.p. 418–420 K, $[\alpha]_D^{25{\cdot}0°C} = -22{\cdot}15°$ [dimethylformamide, 1 g dm^{-3}].



* Kainic acid is 2-carboxy-4-isopropenyl-3-pyrrolidineacetic acid.

Prismatic crystal 0·20 × 0·56 × 0·78 mm; Enraf–Nonius CAD-4 diffractometer, ω–2θ scan technique for data collection; unit cell from LS fit on 25 reflections in range $7 < \theta < 10°$; 8034 measured reflections up to $2\theta_{max} = 56°$ in index range $-11 < h < 11$, $0 < k < 18$, $0 < l < 22$, averaged 3681; ω-scan width $(0{\cdot}8 + 0{\cdot}3\tan\theta)°$, scan rate 1·18–5·49° min^{-1}, max. scan time 60 s, horizontal counter aperture $(2{\cdot}4 + 0{\cdot}9\tan\theta)$ mm; three reference reflections every 2 h, intensity decrease 0·4%, three orientation-control reflections after every 400 reflections, 1514 unique observed reflections with $I > 2\sigma(I)$ used for analysis. Final $R = 0{\cdot}044$, $wR = 0{\cdot}046$, $\sum w(\Delta F)^2$ minimized, $w = k/[\sigma^2(F) + 0{\cdot}00200(F)^2]$, max. $\Delta/\sigma < 0{\cdot}24$ in final cycle; residual electron density in final difference synthesis between +0·08 and −0·10 e Å^{-3}; atomic scattering factors from *SHELX*76 (Sheldrick, 1976); no absorption correction applied; all non-H atoms refined with anisotropic temperature factors; H atoms calculated with riding model (C—H = 1·08 Å, $B_{iso} = 9{\cdot}65$ Å^2), except H9*A*, H9*B*, H10, H11, H12, H15*A*, H15*B* and H18 which were located in difference syntheses and refined isotropically. Computer programs used: *PATSEE* (Egert, 1985) and *SHELXS*86 (Sheldrick, 1986) for the solution of the structure, *SHELX*76 (Sheldrick, 1976) and *PLUTO* (Motherwell & Clegg,

0108-2701/89/101649-03$03.00

Table 1. *Atomic positional* ($\times 10^4$, *for* H $\times 10^3$) *and isotropic thermal parameters* ($Å^2$)

$$B_{eq} = \tfrac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	*y*	*z*	B_{eq}
C1	1446 (7)	2873 (4)	4203 (3)	4·72 (19)
C2	2858 (9)	3251 (5)	3835 (4)	7·74 (25)
C3	315 (9)	2488 (4)	3714 (3)	6·41 (21)
C4	1941 (10)	2228 (4)	4743 (3)	6·92 (22)
O5	588 (4)	3587 (2)	4495 (2)	4·68 (11)
C6	1192 (7)	4146 (4)	4934 (3)	4·15 (16)
O7	2398 (4)	4026 (3)	5249 (2)	4·95 (12)
N8	319 (5)	4847 (3)	5001 (2)	3·69 (11)
C9	702 (8)	5584 (5)	5419 (4)	4·51 (22)
C10	−255 (6)	6316 (4)	5150 (3)	3·84 (17)
C11	−1748 (5)	5835 (4)	4927 (3)	3·52 (14)
C12	−1079 (6)	5005 (4)	4594 (3)	3·67 (16)
C13	−497 (7)	7099 (4)	5576 (4)	5·46 (20)
C14	−1492 (9)	7789 (4)	5272 (4)	7·23 (24)
C15	140 (10)	7175 (6)	6187 (4)	8·12 (28)
C16	−2830 (6)	5602 (4)	5498 (3)	4·19 (16)
C17	−4296 (6)	5192 (4)	5242 (3)	4·19 (16)
O18	−5073 (6)	4820 (4)	5719 (2)	5·79 (15)
O19	−4692 (5)	5188 (3)	4672 (2)	6·93 (15)
C20	−699 (6)	5148 (4)	3887 (3)	3·90 (16)
O21	457 (5)	5453 (3)	3674 (2)	5·99 (12)
O22	−1913 (4)	4939 (3)	3510 (2)	4·92 (12)
C23	−1835 (7)	5094 (4)	2788 (3)	4·61 (18)
C24	−3419 (7)	5386 (4)	2584 (3)	4·30 (17)
C25	−4721 (8)	5277 (5)	2953 (3)	6·18 (20)
C26	−6153 (8)	5536 (5)	2716 (4)	7·02 (26)
C27	−6292 (8)	5918 (5)	2113 (4)	6·49 (24)
C28	−4966 (9)	6042 (5)	1719 (3)	6·30 (23)
C29	−3572 (8)	5779 (4)	1968 (3)	5·25 (20)
C30	−1288 (7)	4285 (4)	2457 (3)	5·05 (19)
C31	−2213 (10)	3567 (5)	2390 (4)	7·00 (25)
C32	−1633 (14)	2796 (6)	2073 (4)	8·40 (34)
C33	−147 (18)	2799 (8)	1846 (4)	10·21 (50)
C34	700 (13)	3498 (8)	1884 (5)	9·90 (37)
C35	178 (9)	4251 (6)	2191 (3)	7·22 (25)
				B_{iso}
H9*A*	57 (6)	546 (4)	594 (3)	5·4 (1·4)
H9*B*	141 (5)	569 (3)	540 (2)	1·3 (1·2)
H10	32 (6)	653 (3)	474 (3)	5·2 (1·3)
H11	−219 (5)	613 (3)	454 (2)	3·3 (1·1)
H12	−178 (6)	452 (3)	459 (2)	3·3 (1·1)
H15*A*	−28 (7)	773 (4)	648 (3)	5·8 (1·4)
H15*B*	117 (11)	680 (5)	636 (4)	10·6 (2·4)
H18	−573 (9)	459 (5)	570 (4)	5·7 (2·3)

Table 2. *Selected bond distances* (Å) *and angles* (°)

O7—C6	1·239 (6)	C12—N8—C9	112·3 (5)
C9—N8	1·451 (8)	C10—C9—N8	104·5 (5)
C12—N8	1·485 (6)	C11—C10—C9	102·0 (5)
C10—C9	1·502 (9)	C12—C11—C10	101·7 (4)
C11—C10	1·560 (7)	C11—C12—N8	101·7 (4)
C12—C11	1·555 (7)		
C15—C13	1·354 (9)	C6—N8—C12—C20	−66·7 (6)
O18—C17	1·306 (7)		
O19—C17	1·198 (7)		
O21—C20	1·190 (6)		

Donor—H	Donor···Acceptor	H···Acceptor	Donor—H···Acceptor
O18—H18	O18···O7^{i}	H18···O7	O18—H18···O7
0·673 (75)	2·688 (6)	2·054 (74)	157 (8)

Symmetry code: (i) $x + 1, y, z$.

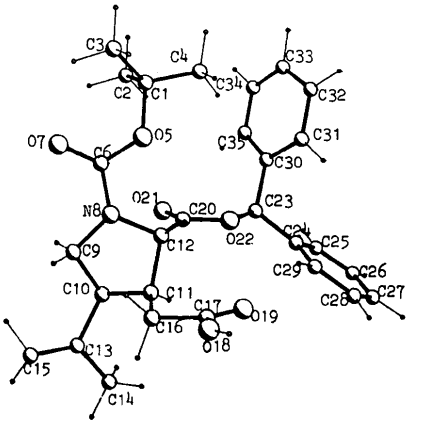


Fig. 1. Perspective view of the molecule with the atomic numbering scheme.

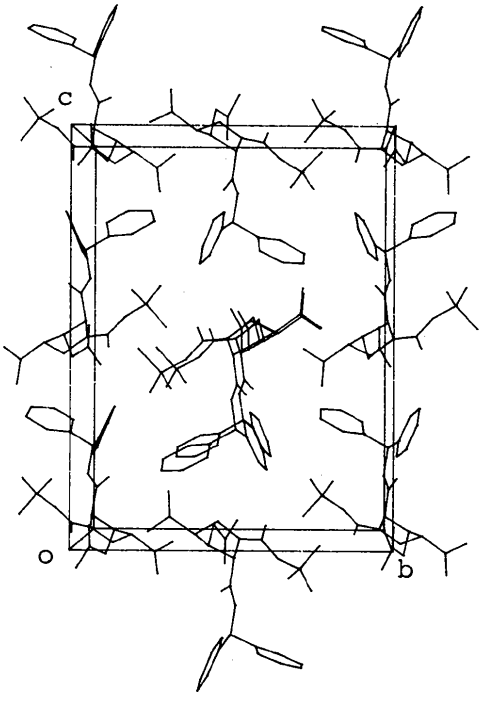


Fig. 2. A view illustrating the packing in the crystal viewed in the direction of the *a* axis.

1978). Atomic positional and equivalent isotropic thermal parameters are presented in Table 1.* Intramolecular bond distances and angles are summarized in Table 2. A view of the molecule with the atom-numbering scheme is shown in Fig. 1. The arrangement of the molecules in the crystal is depicted in Fig. 2.

Related literature. The title compound was prepared according to the method of Goldberg & Teichberg (1985) for the regiospecific synthesis of the corresponding 2-methyl ester. Esterification at the 2-position was accomplished using the Mitsunobu reaction (see *e.g.* Barlos, Kallitsis, Mamos, Patrianakou & Stavropoulos, 1987). The molecule is

* Lists of structure factors, anisotropic thermal parameters, bond distances and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51888 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

an intermediate in the synthesis of γ-dipeptides of the neuroexcitatory amino acid kainic acid.

We thank the Greek Secretariat of Research and Technology for financial support.

References

BARLOS, K., KALLITSIS, J., MAMOS, P., PATRIANAKOU, S. & STAVROPOULOS, G. (1987). *Justus Liebigs Ann. Chem.* pp. 633–635.

EGERT, E. (1985). *PATSEE*. Fragment search by integrated Patterson and direct methods. Univ. of Göttingen, Federal Republic of Germany.

GOLDBERG, O. & TEICHBERG, V. I. (1985). *J. Med. Chem.* **28**, 1957–1958.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1986). *SHELXS86*. Program for crystal structure solution. Univ. of Göttingen, Federal Republic of Germany.

Acta Cryst. (1989). C**45**, 1651–1652

Structure of *N*-Triphenylmethyl-2-oxa-5-azabicyclo[2.2.1]heptan-3-one

BY D. PAPAIOANNOU, G. STAVROPOULOS, V. NASTOPOULOS AND S. VOLIOTIS

Department of Chemistry, University of Patras, GR-26110 Patras, Greece

AND I. LEBAN

Department of Chemistry and Chemical Technology, Edvard Kardelj University, Murnikova 6, PO Box 537, 61001 Ljubljana, Yugoslavia

(*Received* 15 *December* 1988; *accepted* 4 *April* 1989)

Abstract. $C_{24}H_{21}NO_2$, $M_r = 355{\cdot}42$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}503\ (2)$, $b = 12{\cdot}853\ (3)$, $c = 15{\cdot}601\ (4)$ Å, $V = 1905{\cdot}5\ (8)$ Å^3, $Z = 4$, $D_x = 1{\cdot}24$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}85$ cm^{-1}, $F(000) = 752$, room temperature, $R = 0{\cdot}039$ for 1079 unique observed reflections. The atoms C(24), N(20), C(21) and C(22) of the proline ring [O$_2$C—C(24)—N(20)—C(21)—C(22)—C(23)] lie in a plane to within ±0·06 Å while C(23) is out of the plane by 0·85 Å. The two planes of the six-membered ring [C(24)—N(20)—C(21)—C(22) and C(24)—C(25)—O(26)—C(22)] form a dihedral angle of 55 (1)°. The dihedral angles between the plane of the three C atoms C(22), C(23) and C(24) and the two planes of the six-membered ring are 126 (1) and 71 (1)° respectively. Other bond lengths and angles are normal.

Experimental. Recrystallization from acetone, m.p. 503–508 K, $[\alpha]_D^{25°C} = +104{\cdot}6°$ [$CHCl_3$, 1 g dm^{-3}]. Plate crystal 0·48 × 0·48 × 0·08 mm, Enraf–Nonius CAD-4 diffractometer, ω–2θ-scan technique for data collection, lattice parameters from 25 reflections in range $7 < \theta < 10°$. Index range $0 < h < 12$, $0 < k < 14$, $-19 < l < 19$ for 5092 measured reflections up to $2\theta_{max} = 54°$. Averaged 2391, mean discrepancy on I 1·7% (for 4890 reflections); ω-scan width (0·7 + 0·3tanθ)°, scan rate 1·03–5·49° min^{-1}; horizontal aperture (2·4 + 0·9tanθ) mm, max. scan time 60 s; intensities of 00$\bar{4}$, 10$\bar{2}$ and 02$\bar{3}$ remeasured every 2 h, intensity decrease 2·5%, orientation-control reflections (13$\bar{4}$, 24$\bar{3}$, 22$\bar{7}$) every 500 reflections. 1079 unique observed reflections with $I > 1{\cdot}5\sigma(I)$ used for refinement. Final $R = 0{\cdot}039$, $wR = 0{\cdot}043$, $\sum w(\Delta F)^2$ minimized, $w = 0{\cdot}1307/[\sigma^2(F) + 0{\cdot}010869(F)^2]$, max. $\Delta/\sigma < 0{\cdot}7$ in final cycle (for non-H atoms); residual electron density in final difference synthesis between +0·12 and −0·18 e Å^{-3}, atomic scattering factors from *SHELX76* (Sheldrick, 1976); no absorption correction applied; heavier atoms refined with anisotropic temperature factors; H atoms positioned geometrically, riding model [C—H = 1·08 Å, overall U(H) = 0·069 (4) Å^2]. Computer programs used: *MULTAN87* (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987), *SHELX76* (Sheldrick, 1976) and *PLUTO* (Motherwell & Clegg, 1978). Atomic parameters are given in Table 1,* bond distances and angles in Table 2. Fig. 1 shows the atom numbering of the molecule.

Related literature. For the synthesis of the title and related compounds see Papaioannou, Stavropoulos & Karagiannis (1988), Bowers-Nemia & Joullie

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, details of least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51889 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/89/101651-02$03.00