glutaramide and glutamine analogs as possible antineoplastic agents is evident from their activities by way of glutamine and/or folic acid antagonism. Glutaramic acid analogs may be regarded as the precursors of glutaramides and may interfere with the biochemical and physiological functions of natural glutaramic acid and/or folic acid or their derivatives by taking their place or by blocking the enzymes or coenzymes involved in their metabolism (Debnath, Jha, Majumdar & De, 1987). The X-ray structural information gained from a study of 3-(4-bromophenyl)-5-*n*-butylglutaramic acid may help to reveal the effects of different substituents on antineoplastic activity. The bond distance $C(1)—Br(1) = 1.884 (10)$ Å is quite close to the value of 1.911 Å obtained by Kosuge, Tsuji & Hirai (1981). The deviation of the Br(1) atom from the least-squares plane through the benzenoid ring [C(1)—C(6)] is -0.017 Å. Some intramolecular contacts are $O(1)\cdots O(2)$, 2.209 (13) and $C(12)\cdots O(3)$, 2.833 (18) Å [with an $O\cdots H$ contact of 2.41 (17) Å]. The molecules are linked through hydrogen bonds: $N(1)—H(N1)\cdots O(1)$, $N\cdots O = 2.968$ (16) Å and $O(2)—H(O2)\cdots O(3)$, $O\cdots O = 2.570$ (11) Å.

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Structure of N^2-*p*-Bromophenyl-N^1-methyl-N^1-phenylbenzamidine

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Abstract. $C_{20}H_{17}BrN_2$, $M_r = 365.3$, orthorhombic, $P2_12_12_1$, $a = 9.267$ (1), $b = 12.312$ (1), $c = 15.392$ (1) Å, $V = 1754.6$ (6) Å^3, $Z = 4$, $D_m = 1.36$, $D_x = 1.38$ Mg m^{-3}, $\lambda(Cu\,K\alpha) = 1.54178$ Å, $\mu(Cu\,K\alpha) = 2.9$ mm^{-1}, $F(000) = 744$, room temperature, $R = 0.027$ for 1248 observed reflexions. Crystals of the title compound are isostructural with those of N^1-methyl-N^1-phenyl-N^2-(*p*-tolyl)benzamidine [Oszczapowicz, Tykarska, Jaskólski & Kosturkiewicz (1986). *Acta Cryst.* C**42,** 1816–1818], which suggests that a Br atom exerts the same steric effect as a methyl group, and which confirms the stereochemical similarity between these two molecules. In both molecules the phenyl rings are situated on one side of the amidine moiety while the other side is occupied by the methyl group alone, and the configuration around the double bond $C—N^2$ is *trans* (*E*). The steric hindrance is primarily relaxed by twisting of the phenyl substituents at N^1, $C_{amidine}$ and N^2 relative to the central amidine plane by 62.2 (5), 60.6 (5) and 71.1 (5)°, respectively. Two $C—N$ bonds in the amidine moiety differ in length [1.359 (5) and 1.291 (5) Å for $C—N^1$ and $C—N^2$, respectively] and the $N^1—C—N^2$ angle is 118.9 (3)°. These values are similar to those found in the *p*-tolyl derivative.

Experimental. The title compound was synthesized by Oszczapowicz, Raczyńska & Orliński (1981).

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_k U_{ik}a_i^*a_k^*(\mathbf{a}_i.\mathbf{a}_k).$$

	x	y	z	U_{eq}
Br(1)	0·35248 (7)	−0·15007 (5)	−0·26524 (4)	0·0907 (2)
N(1)	0·5303 (4)	0·2455 (2)	0·1503 (2)	0·049 (1)
N(2)	0·4132 (4)	0·1673 (3)	0·0351 (2)	0·054 (1)
C(1)	0·5144 (4)	0·1617 (3)	0·0926 (2)	0·044 (1)
C(2)	0·6195 (4)	0·0705 (3)	0·0992 (2)	0·041 (1)
C(3)	0·7665 (5)	0·0893 (4)	0·0872 (3)	0·052 (2)
C(4)	0·8628 (5)	0·0025 (4)	0·0907 (3)	0·063 (2)
C(5)	0·8128 (6)	−0·1018 (4)	0·1057 (3)	0·068 (2)
C(6)	0·6681 (6)	−0·1199 (3)	0·1177 (3)	0·063 (2)
C(7)	0·5722 (5)	−0·0348 (3)	0·1136 (3)	0·053 (2)
C(11)	0·6085 (4)	0·2360 (3)	0·2294 (3)	0·045 (1)
C(12)	0·5826 (5)	0·1512 (3)	0·2869 (2)	0·053 (1)
C(13)	0·6611 (6)	0·1462 (4)	0·3633 (3)	0·071 (2)
C(14)	0·7626 (6)	0·2240 (5)	0·3842 (3)	0·077 (2)
C(15)	0·7867 (5)	0·3075 (4)	0·3268 (3)	0·072 (2)
C(16)	0·7111 (5)	0·3132 (4)	0·2491 (3)	0·060 (2)
C(17)	0·4429 (5)	0·3434 (3)	0·1380 (3)	0·064 (2)
C(21)	0·4063 (4)	0·0884 (3)	−0·0319 (3)	0·049 (1)
C(22)	0·2893 (5)	0·0185 (4)	−0·0357 (3)	0·058 (2)
C(23)	0·2769 (5)	−0·0544 (4)	−0·1038 (3)	0·064 (2)
C(24)	0·3790 (5)	−0·0563 (3)	−0·1684 (3)	0·057 (2)
C(25)	0·4954 (5)	0·0120 (3)	−0·1663 (3)	0·058 (2)
C(26)	0·5101 (5)	0·0855 (3)	−0·0983 (3)	0·055 (2)

Table 2. *Bond lengths* (Å) *and angles* (°)

N(1)—C(1)	1·369 (5)	C(13)—C(14)	1·380 (8)
C(1)—N(2)	1·291 (5)	C(14)—C(15)	1·374 (7)
C(1)—C(2)	1·491 (6)	C(15)—C(16)	1·388 (7)
C(2)—C(3)	1·394 (6)	C(16)—C(11)	1·378 (6)
C(3)—C(4)	1·393 (6)	N(2)—C(21)	1·419 (5)
C(4)—C(5)	1·385 (7)	C(21)—C(22)	1·386 (6)
C(5)—C(6)	1·372 (7)	C(22)—C(23)	1·385 (7)
C(6)—C(7)	1·375 (6)	C(23)—C(24)	1·373 (7)
C(7)—C(2)	1·386 (6)	C(24)—C(25)	1·369 (7)
N(1)—C(17)	1·465 (6)	C(25)—C(26)	1·391 (6)
N(1)—C(11)	1·422 (5)	C(26)—C(21)	1·403 (6)
C(11)—C(12)	1·389 (6)	C(24)—Br	1·901 (4)
C(12)—C(13)	1·384 (6)		

N(1)—C(1)—N(2)	118·9 (3)	C(14)—C(15)—C(16)	120·6 (4)
N(1)—C(1)—C(2)	117·0 (3)	C(15)—C(16)—C(11)	120·2 (4)
C(1)—N(1)—C(17)	118·5 (3)	C(12)—C(11)—C(16)	119·8 (3)
C(1)—N(1)—C(11)	123·2 (3)	N(1)—C(11)—C(16)	118·9 (3)
N(2)—C(1)—C(2)	124·2 (3)	C(11)—N(1)—C(17)	117·4 (3)
C(1)—C(2)—C(3)	120·3 (3)	C(1)—N(2)—C(21)	119·7 (3)
C(2)—C(3)—C(4)	119·6 (4)	N(2)—C(21)—C(22)	119·4 (3)
C(3)—C(4)—C(5)	120·2 (4)	N(2)—C(21)—C(26)	121·1 (3)
C(4)—C(5)—C(6)	120·0 (4)	C(21)—C(22)—C(23)	120·0 (4)
C(5)—C(6)—C(7)	120·1 (4)	C(22)—C(23)—C(24)	120·2 (4)
C(6)—C(7)—C(2)	121·0 (4)	C(23)—C(24)—C(25)	121·0 (4)
C(7)—C(2)—C(3)	119·1 (3)	C(24)—C(25)—C(26)	119·7 (4)
C(7)—C(2)—C(1)	120·6 (3)	C(21)—C(26)—C(25)	119·9 (4)
N(1)—C(11)—C(12)	121·3 (3)	C(26)—C(21)—C(22)	119·3 (3)
C(11)—C(12)—C(13)	118·9 (3)	C(22)—C(21)—N(2)	119·4 (3)
C(12)—C(13)—C(14)	121·7 (4)	C(23)—C(24)—Br	119·3 (3)
C(13)—C(14)—C(15)	118·7 (4)		

Crystal obtained from anhydrous acetone/hexane solution; density by flotation. Space group from Weissenberg photographs. Crystal 0·32 × 0·30 × 0·25 mm, Syntex $P2_1$ diffractometer, cell parameters from least-squares treatment of setting angles of 15 reflexions with $16 \le 2\theta \le 22°$. 1838 reflexions with $2\theta \le 115°$ measured in range $h\ 0\rightarrow11$, $k\ 0\rightarrow14$, $l\ 0\rightarrow$

18. No significant intensity variation ($\pm 1·1$%) for two standard reflexions (020 and $\bar{2}12$) recorded every 100 reflexions. Peak-profile analysis according to Lehmann & Larsen (1974); no absorption correction. 1252 observed reflexions with $I \ge 2\sigma(I)$. Structure solved by heavy-atom method and refined by full-matrix least-squares calculations on F's, initially with isotropic and finally with anisotropic temperature factors for the non-H atoms. Function minimized: $w(F_o - F_c)^2$, with weights based on counting statistics: $w = 1/\sigma(F_o)$. H-atom positions from molecular geometry, assigned isotropic temperature factor $U = 0·07$ Å^2, allowed to ride on parent C atoms, methyl group treated as rigid body, H atoms included as fixed isotropic contribution to F_c. Empirical extinction parameter x refined to $2·0 \times 10^{-6}$ and F_c multiplied by $(1 - xF_c^2)/\sin\theta$. Four reflexions (601, 052, 075, 092) with large $\Delta F/\sigma$ excluded from final refinement. Refinement converged with $R = 0·027$, $wR = 0·031$, $S = 3·54$. Max. (Δ/σ) in the last cycle = 0·07, $(\Delta\rho)_{max} = 0·20$, $(\Delta\rho)_{min} = -0·27$ e Å^{-3}. Scattering factors as in *SHELX*76 (Sheldrick, 1976). Computations by *SHELX*76 and local programs (Jaskólski, 1982). In the final stages of refinement additional cycles were performed to differentiate between two enantiomeric structures. Changing the signs of the atomic positional parameters resulted in convergence at $R = 0·038$ and $wR = 0·046$, indicating the correctness of the originally chosen structure.

The molecular structure with the atomic labelling is shown in Fig. 1. Positional parameters and the equivalent U values are given in Table 1.* Bond lengths and angles are presented in Table 2.

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* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51920 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
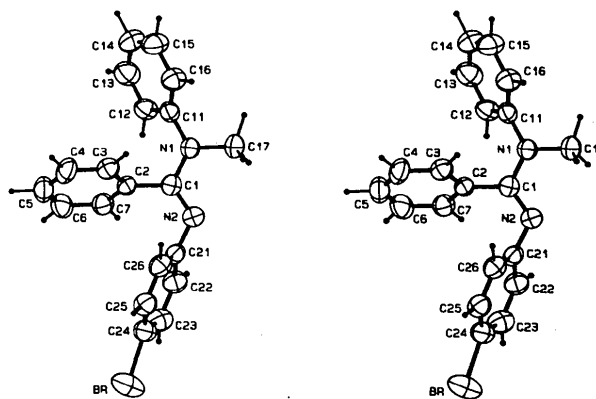

Fig. 1. Stereoview of the title molecule. H atoms are represented by dots.

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).

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Structure of N-(tert-Butoxycarbonyl)kainic Acid 2-Diphenylmethyl Ester*
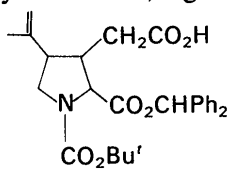
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Abstract. $C_{28}H_{33}NO_6$, $M_r = 479.54$, orthorhombic, $P2_12_12_1$, $a = 8.694$ (2), $b = 15.397$ (3), $c = 20.120$ (4) Å, $V = 2693.3$ (9) Å^3, $Z = 4$, $D_x = 1.183$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 0.77$ cm^{-1}, $F(000) = 1024$, room temperature, $R = 0.044$ for 1514 unique observed reflections. The atoms N8, C9, C11 and C12 of the proline ring (O$_2$C—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, $[\alpha]_D^{25 \cdot 0°C} = -22.15°$ [dimethylformamide, 1 g dm^{-3}].

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

Prismatic crystal $0.20 \times 0.56 \times 0.78$ 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 $(0.8 + 0.3\tan\theta)°$, scan rate 1.18–$5.49°$ min^{-1}, max. scan time 60 s, horizontal counter aperture $(2.4 + 0.9\tan\theta)$ 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 $R = 0.044$, $wR = 0.046$, $\Sigma w(\Delta F)^2$ minimized, $w = k/[\sigma^2(F) + 0.00200(F)^2]$, max. $\Delta/\sigma < 0.24$ in final cycle; residual electron density in final difference synthesis between $+0.08$ and -0.10 e Å^{-3}; atomic scattering factors from *SHELX76* (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, $B_{iso} = 9.65$ Å^2), except H9A, H9B, H10, H11, H12, H15A, H15B and H18 which were located in difference syntheses and refined isotropically. Computer programs used: *PATSEE* (Egert, 1985) and *SHELXS86* (Sheldrick, 1986) for the solution of the structure, *SHELX76* (Sheldrick, 1976) and *PLUTO* (Motherwell & Clegg,