carboxylate group in the present crystal is typical of the dissociated one. The guanine moieties are almost planar, the maximum deviation of atoms from the least-squares plane being 0·048 Å. The conformations of the carboxyethyl groups are nearly the same for the two independent molecules; the torsion angle N(9)–C(10)–C(11)–C(12) is −63·2 (7)° for one molecule and 63·9 (8)° for the other. The bond distances and angles of the 1,6-hexanediamines are similar to those in 1,6-hexanediamine (Binnie & Robertson, 1950). The molecule has a *trans* zigzag conformation, slightly twisted around the C(1′)–C(2′) and C(2′)–C(3′) bonds.

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Structure of *cyclo*(L-Phenylalanyl-L-phenylalanyl-)

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Abstract. 3,6-Bis(phenylmethyl)-2,5-piperazinedione, $C_{18}H_{18}N_2O_2$, $M_r = 294{\cdot}3$, orthorhombic, $P22_12_1$, $a = 6{\cdot}181$ (1), $b = 10{\cdot}380$ (3), $c = 23{\cdot}795$ (4) Å, $V = 1526{\cdot}8$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}28$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 6{\cdot}0$ cm^{-1}, $F(000) = 624$, room temperature, $R = 0{\cdot}056$ for 959 observed reflections and 176 variables. The 2,5-piperazinedione ring has a flattened boat conformation with the C^β atoms in pseudoaxial positions. One of the phenylalanyl residues faces the 2,5-piperazinedione ring [$\chi_1^1 = 69{\cdot}0$ (6)°], the other is in an extended conformation [$\chi_2^1 = -61{\cdot}9$ (6)°]. The ω torsion angles in both *cis* peptide units are small and equal to −0·9 (7) and 0·1 (7)°.

Introduction. The preference for an aromatic part of an amino-acid residue to fold over the 2,5-piperazinedione (hereafter DKP) skeleton is a well known phenomenon. The folded conformation has been observed both in the solid state and in solution. However, for *cis* cyclic dipeptides built up of two aromatic amino-acid residues, steric repulsions prevent the molecule from adopting a conformation in which both aromatic rings fold over the DKP nucleus. From chirooptical properties (Edelhoch, Bernstein & Wilchek, 1968; Strickland, Wilchek, Horwitz & Billups, 1970) and ^{1}H NMR, ^{13}C NMR spectra (Deslauries, Grzonka, Schaumburg, Shiba & Walter, 1975; Kopple & Marr, 1967) of *cyclo*(L-*X*)$_2$ where *X* is an aromatic amino-acid residue, a conformation with both aromatic rings sharing the space over the DKP ring in a 'face-to-face' fashion was postulated as the most preferred one. On the other hand, the conformational-energy calculations for *cyclo*(L-Tyr)$_2$ (Snow, Hooker & Schellman, 1977), ^{1}H NMR studies of specifically labeled *cyclo*(L-Phe-L-*X*) (Liberek & Bednarek, 1978) and the results of X-ray analaysis of *cyclo*(*N*-Me-L-Phe)$_2$ (Benedetti, Marsh & Goodman, 1976) indicate that the most stable conformation is that in which one of the amino-acid residues is in the folded and the other in the extended conformation.

The present crystal-structure analysis of *cyclo*-(L-Phe)$_2$ has been performed to provide more data on cyclic dipeptides with two aromatic amino-acid residues.

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Experimental. Crystals obtained by sublimation under reduced pressure; crystal approximately 0·02 × 0·1 × 0·5 mm used for measurements, Syntex $P2_1$ diffractometer, graphite monochromator, lattice parameters from 15 reflections with $20 < 2\theta < 38°$, profiles measured for 1281 reflections with $2\theta \leq 115°$ (h 0→6, k 0→11, l 0→25), ω–2θ scan technique, constant scan rate 1° min^{-1}, profile analysis according to Lehmann & Larsen (1974), no significant intensity variation for two standard reflections, absorption ignored, 959 reflections with $I \geq 1{\cdot}96\sigma(I)$; structure solved using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), solution straightforward using default parameters; full-matrix least-squares refinement on F with *SHELX*76 (Sheldrick, 1976), unit weights, phenyl groups treated as regular hexagons (bond lengths in hexagon 1·395 Å) and refined as rigid groups, positions of H atoms bonded to C atoms calculated from geometrical conditions (C—H distances 1·08 Å), H atoms connected to N atoms located on ΔF map.

Final refinement cycle with all non-H atoms anisotropic, rigid phenyl groups, fixed positional and isotropic thermal parameters of H atoms converged to $R = 0{\cdot}056$, $wR = 0{\cdot}052$ for 959 observed reflections and 176 parameters. Max. Δ/σ in last least-squares cycle <0·01. Final difference Fourier map showed no peaks higher than 0·17 e Å^{-3} and lower than −0·20 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computer programs used: *SHELX*76 (Sheldrick, 1976), local programs (Jaskólski, 1982), *PLUTO* (Motherwell & Clegg, 1978).

Discussion. Final positional parameters are given in Table 1, molecular dimensions in Table 2.*

Fig. 1 shows the *cyclo*(L-Phe)$_2$ molecule with the numbering scheme. The atoms C(*A*1), C(*P*1), N(2), C(*A*2) from one peptide unit and C(*A*2), C(*P*2), N(1), C(*A*1) from the second peptide unit present in the molecule are coplanar as shown by the small values of the $\omega_1 = -0{\cdot}9$ (7) and $\omega_2 = 0{\cdot}1$ (7)° torsion angles (torsion angles according to IUPAC–IUB Commission on Biochemical Nomenclature, 1970). The Hooker β parameter (Hooker, Bayley, Radding & Schellman, 1974), which describes the conformation of DKP rings, amounts to −11·0 (8) and indicates a flattened boat with the C^β atoms in pseudoaxial positions. The C^α atoms of the phenylalanyl residues deviate from the mean plane passing through the remaining atoms of the DKP ring by 0·113 (7) and 0·124 (6) Å. According to the nomenclature proposed by Young, Madison & Blout (1976), one of the phenylalanyl side chains has an extended to nitrogen (E_N) conformation as shown by the χ_2^1 and $\chi_2^{2,2}$ torsion angles [−61·9 (6) and 94·8 (5)°, respectively] and the other has a folded (F) conformation [$\chi_1^1 = 69{\cdot}0$ (6), $\chi_1^{2,2} = 91{\cdot}6$ (6)°]. The present conformation of the *cyclo*(L-Phe)$_2$ molecule agrees very well with the minimum-energy conformation found by Snow *et al.* (1977) for *cyclo*(L-Tyr)$_2$.

The molecular packing is shown in Fig. 2. Each dipeptide molecule forms a pair of N—H···O hydrogen bonds [O(1)···N(1^i) 2·891 (7), N(2)···O(2^i) 2·931 (7) Å; (i): 1 + x, y, z] to an adjacent a-translated molecule. This crystal packing corresponds to that predicted by Benedetti, Corradini & Pedone (1969) for diketopiperazines in the solid state.

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

$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$.

	x	y	z	U_{eq}
O(1)	1·1285 (7)	0·4342 (4)	0·3099 (2)	0·052 (2)
O(2)	0·4152 (8)	0·1542 (5)	0·2755 (2)	0·064 (2)
N(1)	0·5678 (9)	0·3469 (5)	0·2928 (2)	0·049 (2)
N(2)	0·9738 (8)	0·2389 (5)	0·3013 (2)	0·043 (2)
C(*P*1)	0·964 (1)	0·3664 (6)	0·3064 (3)	0·047 (2)
C(*P*2)	0·577 (1)	0·2186 (6)	0·2874 (3)	0·048 (2)
C(*A*1)	0·746 (1)	0·4315 (6)	0·3070 (3)	0·048 (2)
C(*A*2)	0·788 (1)	0·1512 (6)	0·2966 (3)	0·044 (2)
C(*B*1)	0·703 (1)	0·4996 (6)	0·3640 (3)	0·058 (2)
C(*B*2)	0·762 (1)	0·0662 (6)	0·3501 (3)	0·054 (2)
C(11)	0·5254 (7)	0·3414 (5)	0·4305 (2)	0·071 (3)
C(21)	0·5349 (7)	0·2555 (5)	0·4755 (2)	0·087 (4)
C(31)	0·7290 (7)	0·2374 (5)	0·5044 (2)	0·094 (4)
C(41)	0·9137 (7)	0·3052 (5)	0·4882 (2)	0·088 (4)
C(51)	0·9042 (7)	0·3911 (5)	0·4432 (2)	0·070 (3)
C(*G*1)	0·7100 (7)	0·4092 (5)	0·4143 (2)	0·060 (3)
C(12)	0·9625 (6)	−0·1431 (4)	0·3367 (2)	0·058 (3)
C(22)	1·1363 (6)	−0·2248 (4)	0·3481 (2)	0·059 (3)
C(32)	1·2992 (6)	−0·1854 (4)	0·3849 (2)	0·065 (3)
C(42)	1·2883 (6)	−0·0644 (4)	0·4102 (2)	0·062 (3)
C(52)	1·1146 (6)	0·0173 (4)	0·3988 (2)	0·059 (3)
C(*G*2)	0·9517 (6)	−0·0221 (4)	0·3620 (2)	0·049 (2)

Table 2. *Molecular dimensions*

(*a*) Bond lengths (Å)

C(*P*1)–O(1)	1·241 (8)	C(*P*2)–O(2)	1·236 (8)
C(*P*1)–N(2)	1·330 (8)	C(*P*2)–N(1)	1·340 (8)
C(*P*1)–C(*A*1)	1·506 (9)	C(*P*2)–C(*A*2)	1·494 (2)
C(*A*1)–N(1)	1·450 (8)	C(*A*2)–N(2)	1·471 (8)
C(*A*1)–C(*B*1)	1·551 (10)	C(*A*2)–C(*B*2)	1·557 (9)
C(*B*1)–C(*G*1)	1·523 (8)	C(*B*2)–C(*G*2)	1·514 (8)

(*b*) Bond angles (°)

N(2)–C(*P*1)–C(*A*1)	119·3 (5)	N(1)–C(*P*2)–C(*A*2)	119·3 (5)
N(2)–C(*P*1)–O(1)	122·2 (5)	N(1)–C(*P*2)–O(2)	121·7 (5)
C(*A*1)–C(*P*1)–O(1)	118·6 (5)	C(*A*2)–C(*P*2)–O(2)	119·1 (5)
C(*P*1)–C(*A*1)–C(*B*1)	111·5 (5)	C(*P*2)–C(*A*2)–C(*B*2)	107·2 (5)
C(*P*1)–C(*A*1)–N(1)	113·9 (5)	C(*P*2)–C(*A*2)–N(2)	113·7 (5)
C(*B*1)–C(*A*1)–N(1)	110·5 (5)	C(*B*2)–C(*A*2)–N(2)	111·7 (5)
C(*A*1)–N(1)–C(*P*2)	126·3 (5)	C(*A*2)–N(2)–C(*P*1)	125·9 (5)
C(*A*1)–C(*B*1)–C(*G*1)	113·6 (5)	C(*A*2)–C(*B*2)–C(*G*2)	114·6 (5)
C(*B*1)–C(*G*1)–C(11)	120·1 (4)	C(*B*2)–C(*G*2)–C(12)	120·3 (4)
C(*B*1)–C(*G*1)–C(51)	119·9 (4)	C(*B*2)–C(*G*2)–C(52)	119·7 (4)

* Lists of observed and calculated structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42905 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

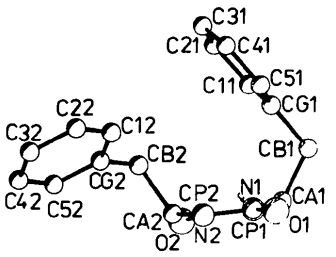

Fig. 1. The conformation of the *cyclo*(L-Phe)$_2$ molecule viewed along N(2)–C(*P*2).

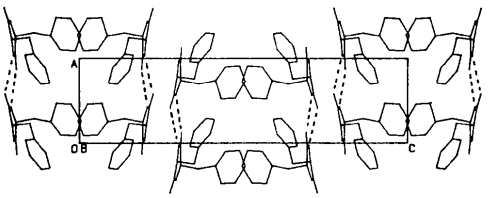

Fig. 2. The crystal structure of *cyclo*(L-Phe)$_2$ viewed down the *b* axis.

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Structure of *N*-[(3*RS*,5*SR*)-1-Benzyl-5-methyl-3-pyrrolidinyl]-5-chloro-2-methoxy-4-methylaminobenzamide Hydrochloride*

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Abstract. $C_{21}H_{27}ClN_3O_2^+.Cl^-$, $M_r = 424{\cdot}37$, monoclinic, $P2_1/c$, $a = 13{\cdot}820\ (3)$, $b = 13{\cdot}002\ (3)$, $c = 12{\cdot}714\ (3)$ Å, $\beta = 100{\cdot}66\ (2)°$, $V = 2245{\cdot}1\ (9)$ Å^3, $Z = 4$, $D_x = 1{\cdot}256$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 28{\cdot}6$ cm^{-1}, $F(000) = 896$, $T = 298$ K, $R = 0{\cdot}062$ for 2222 observed reflections with $|F_o| > 3\sigma(|F_o|)$. An intramolecular H bond between the amide N and the methoxy O is observed. The distance between the tertiary N and the center of the benzene ring is 7·12 Å, and the deviation of the N from the benzene plane is 0·57 Å.

* New Potent Neuroleptic Drugs of Benzamide Derivatives. VI.

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