N,N'-Bis(methoxycarbonylmethyl)-terephthalamide

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Molecules of the title compound, dimethyl N,N'-bis(1,4-benzenedicarboxamido)diacetate, C_{14}H_{16}N_{2}O_{6}, lie on inversion centres and are hydrogen bonded along a single direction that runs parallel to the crystallographic b axis. Glycine residues adopt a conformation which deviates slightly from that characteristic of the polyglycine II structure. An angle close to 27° is found between the planar amide groups and the plane of the aromatic ring.

Comment

Poly(ester amide)s derived from natural amino acids have recently been suggested as a potential family of biodegradable polymers (Paredes, Rodríguez-Galán & Puiggali, 1998; Paredes, Rodríguez-Galán, Puiggali & Peraire, 1998). In order to obtain data for the determination of these polymer structures, different model compounds have been solved (Urpi et al., 1998a,b, 1999). The title compound, (I), has been chosen for the study of polymers derived from terephthalic acid, glycine and different diols, since it may be a model for the common sequence –OCOCH_{2}NHCOCH_{2}CONHCH_{2}COO–. The model molecule is shown in Fig. 1, and selected rotation angles and hydrogen-bond geometry are reported in Tables 1 and 2, respectively.

The amide and ester groups and the benzene ring are planar within experimental accuracy, with root-mean-square distances of the atoms from the best planes defined by them of 0.011, 0.034 and 0.014 Å for C_3/N_1/C_4/O_3/C_5, C_1/O_1/C_2/O_2/C_3 and C_4/C_5/C_6/C_7/C_7'/C_5'/C_4', respectively. The molecule is centrosymmetric and consequently the torsion angles of its two halves are equal but with opposite signs. The glycine residues are characterized by the torsion angles ϕ (C_2–C_3–N_1–C_4) and ψ (O_1–C_2–C_3–N_1), the values of which are very close to those found in the polyglycine II structure (75 and −145°, respectively; Crick & Rich, 1955). The molecular conformation is also characterized by the N_1–C_4–C_5–C_6 torsion angle of 156.09 (13)°, which clearly deviates from 180°. Thus, a displacement of the planar amide group out of the plane of the benzene ring (27°) is produced. This departure from a planar structure (favoured by resonance

Figure 1
A view of (I) with the atom-numbering scheme for the non-H atoms. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as circles with arbitrary radii.

Figure 2
The crystal packing of (I) shown with views normal to (a) the ac and (b) the bc plane. Hydrogen bonds are established along a single direction that runs practically parallel to the a axis.
energy of the conjugate system) can be explained by taking into account a combination of two factors: (i) steric hindrances between the H and O atoms of the amide groups and the nearest H atoms of the aromatic ring; (ii) the establishment in the crystal of intermolecular hydrogen bonds between the amide groups of adjacent molecules. Similar values in the 20–30° interval for the internal rotation angle have been found for different model compounds of aromatic polyamides (Blake & Small, 1972; Palmer & Brisse, 1980; Harkema & Gaymans, 1977) and poly(ester amide)s (Cesari et al., 1976). The molecular packing (Fig. 2) is characterized by the establishment of hydrogen bonds along a single direction. A standard geometry is in agreement with recent calculations on benzene molecules adopt a disposition close to perpendicular, with a distance of 5.13 Å between the centers of the two rings. This geometry is in agreement with recent calculations on benzene dimers (Chipot et al., 1996) that show the T-shaped disposition to be more stable than the stacked one. In the same sense, a T-shaped disposition of aromatic rings seems to be preferred in proteins (Hunter et al., 1991).

**Experimental**

The title compound was synthesized by the reaction of a solution of glycine methyl ester hydrochloride (0.02 mol) and triethylamine (0.04 mol) in chloroform (30 ml) with a solution of terephthaloyl chloride (0.01 mol) in chloroform (20 ml), which was added slowly while maintaining the temperature at 273 K. After 2 h at room temperature, the solution was evaporated yielding a yellow powder that was recrystallized from water (yield 75%, m.p. 435 K). Colorless prismatic crystals were obtained by vapor diffusion (293 K) of a 9:1 (v/v) water/2-propanol solution (concentration 3.6 mg ml⁻¹) against 100% water used as precipitant.

**Crystal data**

\[ C_{14}H_{16}N_{2}O_{6} \]

\[ M_r = 308.29 \]

Monoclinic, \( P2_1/n \)

\[ a = 8.9889 \text{ (10 Å) } \]

\[ b = 4.977 \text{ (2 Å) } \]

\[ c = 16.790 \text{ (4 Å) } \]

\[ \beta = 90.900 \text{ (10°) } \]

\[ V = 751.1 \text{ (4 Å) } ^3 \]

\[ Z = 2 \]

\[ D_r = 1.363 \text{ Mg m}^{-3} \]

Cu Kα radiation

\[ \theta = 8–35° \]

\[ \mu = 0.916 \text{ mm}^{-1} \]

\[ T = 293 \text{ (2 K) } \]

Prism, colourless

\[ 0.20 \times 0.10 \times 0.05 \text{ mm } \]

**Data collection**

Enraf–Nonius CAD-4 diffractometer

\[ \theta_{	ext{max}} = 65.77° \]

\[ h = -10 \rightarrow 10 \]

\[ k = 0 \rightarrow 5 \]

\[ l = -19 \rightarrow 19 \]

2337 measured reflections

1172 independent reflections

1029 reflections with \( I > 2\sigma(I) \)

\[ R_{int} = 0.031 \]

**Refinement**

Refinement on \( F^2 \)

\[ R(F^2) = 0.069 \]

\[ wR(F^2) = 0.175 \]

\[ S = 1.491 \]

1172 reflections

102 parameters

H atoms were placed in calculated positions and refined riding on the atom to which they are attached (N–H = 0.86 Å and C–H = 0.93–0.97 Å), with a fixed isotropic displacement parameter.

Data collection: CAD-4 Software (Kiers, 1994); cell refinement: CAD-4 Software; data reduction: local program; program(s) used to solve structure: SHELXL97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPII (Johnson, 1976).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1152). Services for accessing these data are described at the back of the journal.

**References**


