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N,N'-Bis(methoxycarbonylmethyl)terephthalamide

Elaine Armelin, Encarnación Escudero, Lourdes Campos and Jordi Puiggalí*

Departament d'Enginyeria Química, ETS d'Enginyeria Industrial de Barcelona, Universitat Politècnica de Catalunya, Diagonal 647, Barcelona 08028, Spain Correspondence e-mail: puiggali@eq.upc.es

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Molecules of the title compound, dimethyl N,N'-(1,4-benzenedicarboxamido)diacetate, C₁₄H₁₆N₂O₆, 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 & Puiggalí, 1998; Paredes, Rodríguez-Galán, Puiggalí & Peraire, 1998). In order to obtain data for the determination of these polymer structures, different model compounds have been solved (Urpí *et al.*, 1998*a,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 $-OCOCH_2NHCOC_6H_4CONHCH_2COO-$. The model molecule is shown in Fig. 1, and selected rotation angles and hydrogen-bond geometry are reported in Tables 1 and 2, respectively.

$$CH_3-O-C-CH_2-NH-C$$

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 C3/N1/C4/O3/C5, C1/O1/C2/O2/C3 and C4/C5/C6/C7/C7'/C6'/C5'/C4', 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 (C2–C3–N1–C4) and ψ (O1–C2–C3–N1), the values of which are very close to those found in the polyglycine II





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.

structure (75 and -145° , respectively; Crick & Rich, 1955). The molecular conformation is also characterized by the N1– C4–C5–C6 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 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 found between the hydrogen-bonded molecules, which are not shifted along the molecular axis direction. A twofold screw axis parallel to the b axis relates the non-hydrogen-bonded molecules of the unit cell. The aromatic rings of these two 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 91:9 (ν/ν) water/2-propanol solution (concentration 3.6 mg ml⁻¹) against 100% water used as precipitant.

Crystal data

 $\begin{array}{l} C_{14}H_{16}N_2O_6\\ M_r=308.29\\ \text{Monoclinic, }P2_1/a\\ a=8.9889\ (10)\ \text{\AA}\\ b=4.977\ (2)\ \text{\AA}\\ c=16.790\ (4)\ \text{\AA}\\ \beta=90.900\ (10)^\circ\\ V=751.1\ (4)\ \text{\AA}^3\\ Z=2 \end{array}$

Data collection

Enraf–Nonius CAD-4 diffractometer ω scans 2237 measured reflections 1172 independent reflections 1029 reflections with $I > 2\sigma(I)$ $R_{\rm int} = 0.031$ $D_x = 1.363 \text{ Mg m}^{-3}$ Cu K α radiation Cell parameters from 20 reflections $\theta = 8-35^{\circ}$ $\mu = 0.916 \text{ mm}^{-1}$ T = 293 (2) KPrism, colourless $0.20 \times 0.10 \times 0.05 \text{ mm}$

 $\begin{array}{l} \theta_{\max} = 65.77^{\circ} \\ h = -10 \rightarrow 10 \\ k = 0 \rightarrow 5 \\ l = -19 \rightarrow 19 \\ 3 \text{ standard reflections} \\ \text{frequency: } 60 \text{ min} \\ \text{intensity decay: } 0.5\% \end{array}$

Table 1

Selected torsion angles (°).

N1-C4-C5-C6	156.09 (13)	N1-C3-C2-O1	-152.04 (17)
C4-N1-C3-C2	65.2 (2)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D - \mathbf{H} \cdot \cdot \cdot A$	$D-{\rm H}$	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - \mathbf{H} \cdots A$
$N1-H1\cdots O3^i$	0.86	2.08	2.868 (2)	152

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

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.069$	$w = 1/[\sigma^{\bar{2}}(F_o^2) + (0.1P)^2]$
$wR(F^2) = 0.175$	where $P = (F_o^2 + 2F_c^2)/3$
S = 1.491	$(\Delta/\sigma)_{\rm max} < 0.001$
1172 reflections	$\Delta \rho_{\rm max} = 0.31 \text{ e } \text{\AA}^{-3}$
102 parameters	$\Delta \rho_{\rm min} = -0.29 \text{ e } \text{\AA}^{-3}$

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: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (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.

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