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3-(1-Methyl-1*H*-pyrrole-2-carboxamido)-propanoic acid

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Key indicators

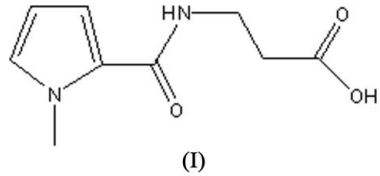
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.123
Data-to-parameter ratio = 15.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_9H_{12}N_2O_3$, was synthesized by condensation of β-alanine methyl ester with 1-methyl-2-(trichloroacetyl)pyrrole at room temperature, followed by saponification and acidification. In the crystal structure, intermolecular N—H···O and O—H···O hydrogen-bond interactions link the molecules into extended ribbons parallel to the a axis.

Comment

Pyrrole derivatives are well known in many marine organisms (Faulkner, 2001); some are bioactive substances (Tasdemir *et al.*, 2002). In our search for bioactive compounds, a series of pyrrole(2-carbonyl)amino acids and their esters, including the title compound, (I), has been synthesized by reaction of amino acid esters with 2-trichloroacetylpyrrole, or 1-methyl-2-trichloroacetylpyrrole, followed by saponification and acidification. Preliminary antibiotic tests performed *in vitro* and determined by the agar dilution method (Feng, 2000) indicate that the title compound inhibits *Streptococcus faecalis* and *Micrococcus luteus*. Antibiotic activities against these two bacteria [determined as minimum inhibitory concentration (mg ml^{-1}) values] are as follows: *Streptococcus faecalis*, 0.156; *Micrococcus luteus*, 0.313. The structure of the title compound, (I), is reported here (Fig. 1).



(I)

Bond lengths and angles are unexceptional and are in good agreement with the corresponding values in 3-[(4-bromo-1-methyl-1*H*-pyrrole-2-ylcarbonyl)amino]propanoic acid (Zeng *et al.*, 2005).

In the crystal structure, there are two types of intermolecular hydrogen bonds (Table 1). Molecules are linked through N—H···O hydrogen bonds to form centrosymmetric dimers (Fig. 2) of graph-set motif $R_2^2(12)$ (Bernstein *et al.*, 1995), and not $R_2^2(8)$ as in alanine (Liao *et al.*, 2001). The dimers are connected by strong O—H···O hydrogen-bond interactions, generating ribbons running parallel to the a axis (Fig. 3).

Experimental

The hydrochloric acid salt of β-alanine methyl ester (0.70 g, 5 mmol) and 1-methyl-2-trichloroacetylpyrrole (1.13 g, 5 mmol) were added to

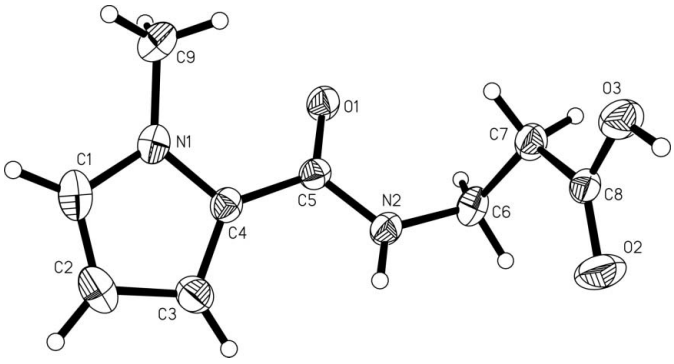


Figure 1
The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids for the non-H atoms are drawn at the 30% probability level.

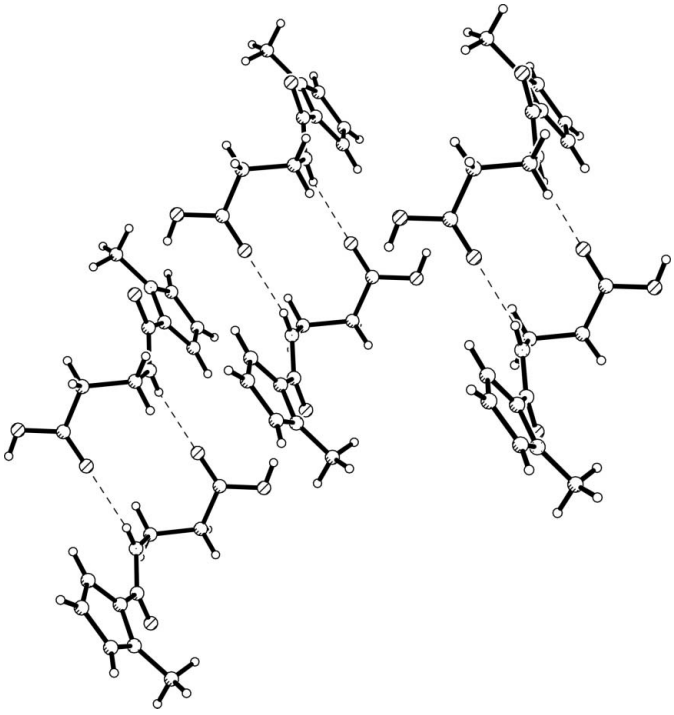


Figure 2
The centrosymmetric dimers formed by hydrogen bonds (dashed lines).

acetonitrile (12 ml), followed by the dropwise addition of triethylamine (1.4 ml). The mixture was stirred at room temperature for 16 h, then poured into water and the yellow product was collected after separating the water. The condensation product was dissolved in 10% aqueous NaOH (10 ml) and ethanol (2 ml), stirred at room temperature for 24 h, then acidified with 10% HCl to pH = 2, and extracted 4 times with 10 ml ethyl acetate. The organic phase was dried with anhydrous sodium sulfate overnight and the solvent removed by distillation under reduced pressure. The pale yellow solid residue was dissolved in ethanol at room temperature. Colorless triclinic crystals suitable for X-ray analysis (m. p. 406 K, in 78.4% yield) grew over a period of 7 days when the solution was exposed to air. ^{1}H NMR (acetone-d_6, 300 Hz): 7.37 (*brs*, 1H), 6.79 (*t*, 1H), 6.68 (*dd*, 1H), 5.97 (*dd*, 1H), 3.89 (*s*, 3H), 3.57–3.50 (*m*, 2H), 2.59 (*t*, 2H); IR (KBr): 3398, 2968, 1716, 1594, 1555, 1515, 1416, 1227; Elemental analysis calculated for $C_9H_{12}N_2O_3$: C 55.09, H 6.16, N 14.28%; found: C 55.23, H 6.10, N 14.12%.

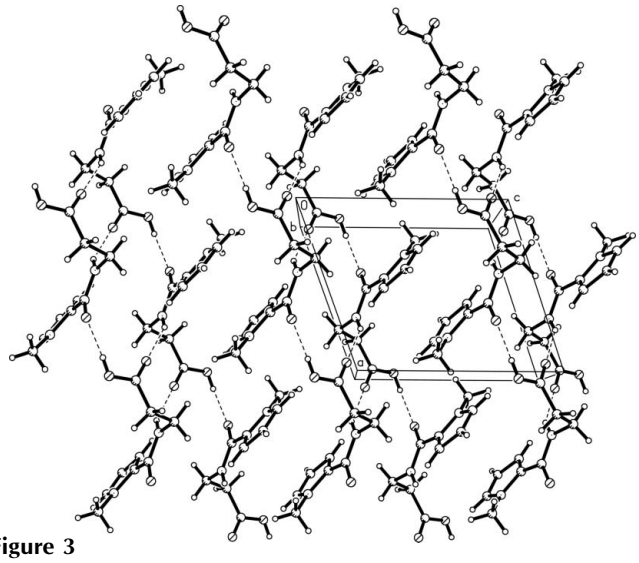


Figure 3
Packing of the title compound, showing the ribbons formed by hydrogen bonds (dashed lines).

Crystal data

$C_9H_{12}N_2O_3$
M_r = 196.21
Triclinic, $P\overline{1}$
a = 7.651 (3) Å
b = 8.270 (3) Å
c = 8.551 (3) Å
α = 88.297 (5)°
β = 70.119 (5)°
γ = 71.488 (5)°
V = 480.7 (3) Å^3
Z = 2
D_x = 1.356 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1580 reflections
θ = 2.6–21.9°
μ = 0.10 mm^{-1}
T = 293 (2) K
Block, colorless
0.40 × 0.35 × 0.19 mm

Data collection

Bruker SMART area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.960, T_{max} = 0.981
3942 measured reflections
2047 independent reflections
1428 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 27.1°
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.123
S = 1.03
2047 reflections
129 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0659P)^2 + 0.0529P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1^{i}	0.82	1.81	2.6176 (18)	167
N2—H2···O2^{ii}	0.86	2.14	2.916 (2)	150

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+2, -y+1, -z$.

All non-H atoms were refined with anisotropic displacement parameters. The H atoms were positioned geometrically (C—H = 0.96 Å for CH_3, C—H = 0.97 Å for CH_2, 0.93 Å for CH, N—H = 0.86 Å and O—H = 0.82 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C,N) (1.5U_{eq} for the methyl group).

Data collection: *SMART* (Bruker,1999); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker 1997); software used to prepare material for publication: *SHELXTL*.

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