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Methyl (4,5-dibromo-1-methyl-1*H*-pyrrole-2-carbonylamino)acetate

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.027
wR factor = 0.068
Data-to-parameter ratio = 18.1

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

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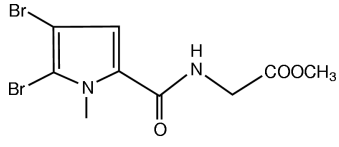
The title compound, $C_9H_{10}Br_2N_2O_3$, was synthesized by chloroform reaction of glycine methyl ester with 4,5-dibromo-1-methyl-2-(trichloroacetyl)pyrrole at room temperature and was obtained in 74.6% yield. The crystal structure was determined by X-ray diffraction.

Comment

Bromopyrrole derivatives are well known in many marine organisms (Faulkner, 2001) and some of them are bioactive substances (Tasdemir *et al.*, 2002). In our search for bioactive compounds, a series of brominated pyrrole-2-carbonylamino acid methyl esters has been synthesized by chloroform reaction, among them the title compound, (I), which was synthesized by reaction of glycine methyl ester with 4,5-dibromo-1-methyl-2-(trichloroacetyl)pyrrole.



(I)

The crystal structure reveals that the title compound has one *N*-methylpyrrole ring and one methyl acetate group linked by an amide function. The supramolecular layers are stabilized by an HN2···O2 hydrogen bond. Pharmacological studies have shown that the title compound possesses moderately antibacterial properties.

Experimental

The hydrochloride of glycine methyl ester (0.63 g, 5 mmol) and 4,5-dibromo-1-methyl-2-(trichloroacetyl)pyrrole (1.92 g, 5 mmol) were added to acetonitrile (12 ml), followed by the dropwise addition of triethylamine (1.4 ml). The mixture was stirred at room temperature

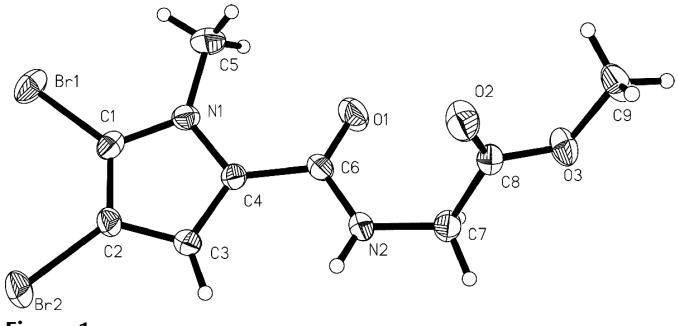


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

for 20 h and then poured into water. After filtration, the precipitate was collected as a yellow solid. The crude product was dissolved in MeOH at room temperature and normal pressure and colorless orthorhombic crystals suitable for X-ray analysis (m.p. 389 K, 74.6% yield) grew over a period of one week when the solution was exposed to the air. IR (KBr): 3390, 3121, 1735, 1658, 1624, 1538, 1500, 1273, 1221 cm^{-1}. Analysis calculated for $C_9H_{10}Br_2N_2O_3$: C 30.54, H 2.85, N 7.91%; found: C 30.85, H 3.02, N 8.08%.

Crystal data

$C_9H_{10}Br_2N_2O_3$
M_r = 354.01
Orthorhombic, $P2_12_12_1$
a = 6.4237 (18) Å
b = 13.445 (4) Å
c = 14.054 (4) Å
V = 1213.8 (6) Å^3
Z = 4
D_x = 1.937 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 907 reflections
θ = 2.9–26.1°
μ = 6.67 mm^{-1}
T = 273 (2) K
Block, colorless
0.50 × 0.30 × 0.17 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.109, T_{max} = 0.322
7341 measured reflections
2621 independent reflections
2257 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.0°
$h = -7 \rightarrow 8$
$k = -8 \rightarrow 17$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.068
S = 1.06
2621 reflections
145 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0323P)^2 + 0.0575P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}
Absolute structure: Flack (1983), 2877 Friedel pairs
Flack parameter = 0.028 (12)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2^{i}	0.86	2.18	2.984 (4)	155

Symmetry code: (i) $x - \frac{1}{2}, \frac{3}{2} - y, 1 - z$.

All H atoms were placed in idealized positions (C—H = 0.96 Å for CH_3, 0.93 Å for CH and 0.86 Å for NH) and refined as riding, with U_{iso}(H) = 1.2 or 1.5 (methyl) times U_{eq}(parent atom). The molecule itself is not chiral but does crystallize in a non-centrosymmetric space group. Since there are heavy atoms in the molecule, the absolute structure can be determined and the Flack (1983) parameter refined to a satisfactory value.

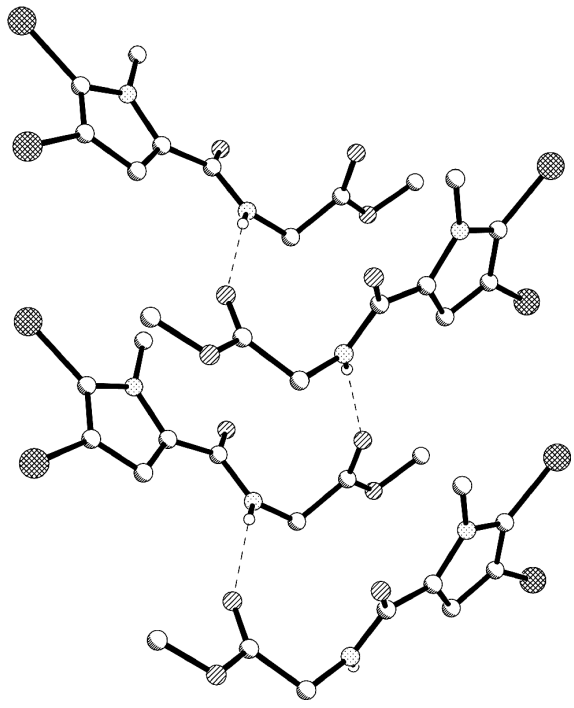


Figure 2
View showing the one-dimensional chain in (I) formed by hydrogen bonds (dashed lines).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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