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# *N*-Methyl-3-(4-bromo-1*H*-pyrrole-2-carbonyl)-aminopropionitrile

**Xiang-Chao Zeng,* Yu-Xia Li, Shi-Hai Xu and Po-Run Liu**

Department of Chemistry, Jinan University, Guangzhou, Guangdong, 510632, People's Republic of China

Correspondence e-mail: xczeng@sohu.com

**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.005 Å
*R* factor = 0.043
*wR* factor = 0.120
Data-to-parameter ratio = 17.8

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

The title compound, $C_9H_{10}BrN_3O$, was synthesized by condensation of *N*-methyl-3-aminopropionitrile with 4-bromo-2-trichloroacetylpyrrole, at room temperature, in 87.3% yield. Intermolecular N—H···O hydrogen bonds give rise to dimers of the compound.

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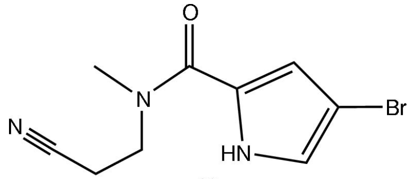
## Comment

Pyrrole derivatives are well known in many marine organisms (Faulkner, 2001), with some of them exhibiting moderate cytotoxicity towards some cancer cells (Konig *et al.*, 1998). In our search for bioactive compounds, a series of *N*-methyl-3-(1*H*-pyrrole-2-carbonyl)aminopropionitriles has been synthesized by reaction of *N*-methyl-3-aminopropionitrile with derivatives of 2-trichloroacetylpyrrole. We report here the crystal structure of the title compound, (I).



(I)

The bond lengths and angles are unexceptional and are in good agreement with corresponding parameters in 3-(4-bromo-1*H*-pyrrole-2-carboxamido)propanoic acid (Zeng *et al.*, 2005) and bis(2-nitriloethyl)aminoacetonitrile (Gao *et al.*, 2004).

In the crystal structure, there is one intermolecular N—H···O hydrogen bond (Table 1), which forms centrosymmetric dimers (Fig. 2) that can be described as $R_2^2(10)$. These

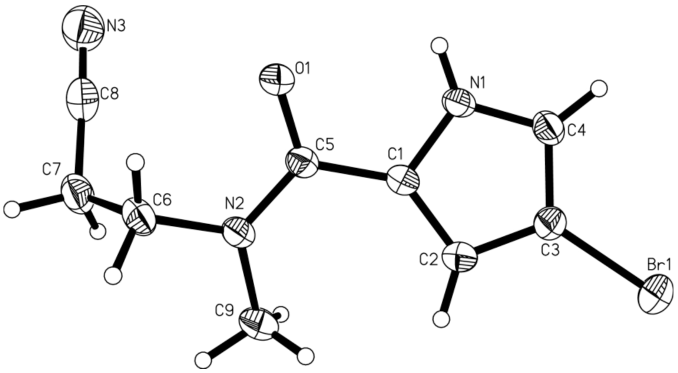


**Figure 1**
The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids for the non-H atoms are drawn at the 30% probability level.

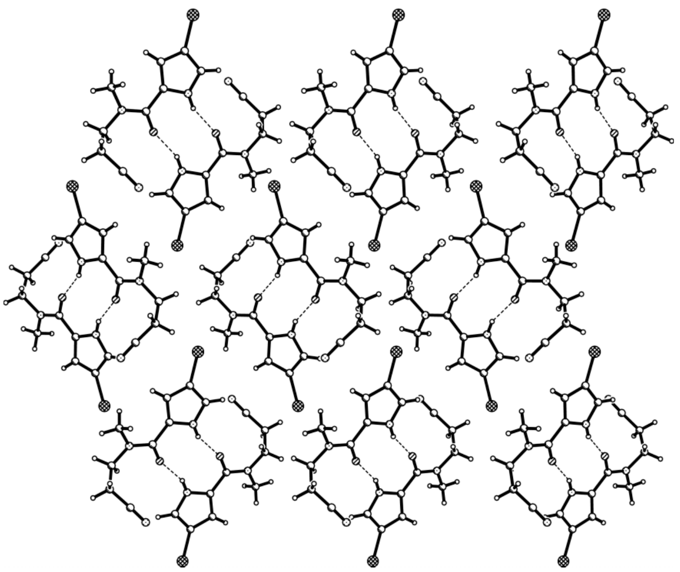


**Figure 2**
View showing the centrosymmetric dimers formed by hydrogen bonds (dashed lines), viewed along the *c* axis.

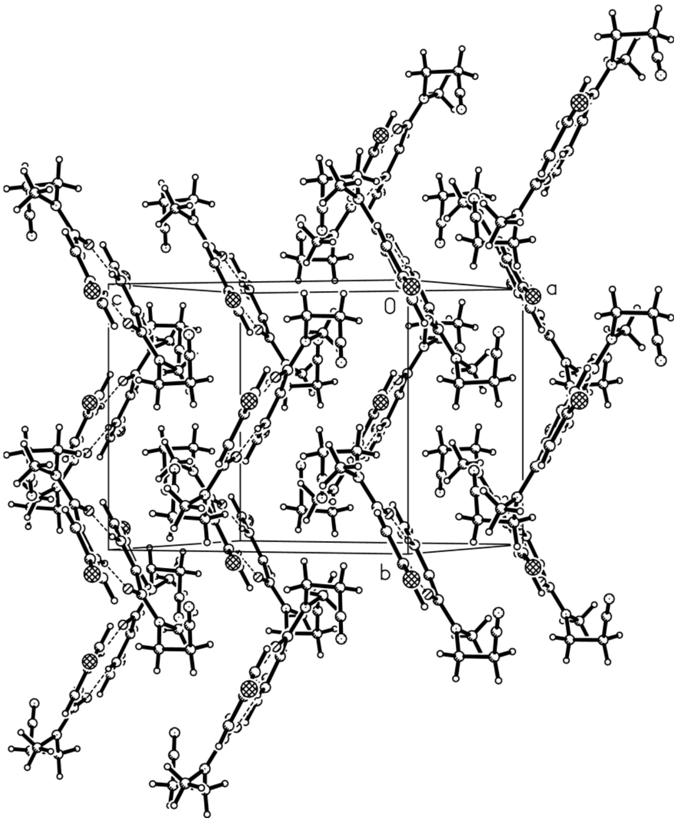


**Figure 3**
The crystal packing. Dashed lines indicate hydrogen bonds.

dimeric units are further assembled into a herringbone pattern in the overall crystal packing (Fig. 3).

## Experimental

*N*-Methyl-3-aminopropionitrile (0.5 ml, 5 mmol) and 4-bromo-2-trichloroacetylpyrrole (1.46 g, 5 mmol) were added to acetonitrile (12 ml). The mixture reacted at room temperature for 16 h and was then poured into water; the brown solid product was collected after filtration. The crude product was dissolved in ethanol–hexane (4:1) at room temperature and pressure. Brown crystals suitable for X-ray analysis (m.p. 397 K, 87.3% yield) grew over a period of one week when the solution was exposed to air. $^1$H NMR: $\delta$ 10.08 (*brs*, 1H), 6.93 (*d*, 1H), 6.60 (*s*, 1H), 3.80 (*t*, 2H), 3.41 (*s*, 3H), 2.72 (*t*, 2H); IR (KBr): 3240, 2930, 2245, 1598, 1546, 1494, 774. Analysis calculated for $C_9H_{10}BrN_3O$: C 42.21, H 3.94, N 16.41%; found: C 42.28, H 4.02, N 16.59%.

### *Crystal data*

$C_9H_{10}BrN_3O$
$M_r = 256.11$
Monoclinic, $P2_1/c$
$a = 8.759\ (2)$ Å
$b = 10.488\ (3)$ Å
$c = 12.080\ (3)$ Å
$\beta = 110.015\ (5)^\circ$
$V = 1042.7\ (5)$ Å$^3$
$Z = 4$

$D_x = 1.632$ Mg m$^{-3}$
Mo *Kα* radiation
Cell parameters from 863 reflections
$\theta = 2.6–26.8^\circ$
$\mu = 3.91$ mm$^{-1}$
$T = 293\ (2)$ K
Parallelepiped, brown
0.50 × 0.36 × 0.28 mm

### *Data collection*

Bruker SMART 1K CCD area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick,1996)
$T_{\min} = 0.122$, $T_{\max} = 0.334$
6363 measured reflections

2262 independent reflections
1741 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.022$
$\theta_{\max} = 27.1^\circ$
$h = -11 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -7 \rightarrow 15$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.120$
$S = 1.05$
2262 reflections
127 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 1.0123P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.99$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.98$ e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O1$^{\text{i}}$ | 0.86 | 2.01 | 2.808 (3) | 155 |

Symmetry code: (i) $-x, -y+1, -z+1$.

H atoms were positioned geometrically (C—H = 0.97 Å for $CH_2$, 0.96 Å for $CH_3$ and 0.93 Å for CH, and N—H = 0.86 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$(parent atom) [$U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C9})$].

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

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