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Key indicators

Single-crystal X-ray study  
 T = 294 K  
 Mean  $\sigma$ (C–C) = 0.003 Å  
 R factor = 0.041  
 wR factor = 0.099  
 Data-to-parameter ratio = 12.2

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

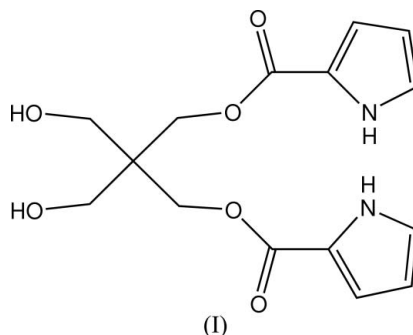
2,2-Bis(hydroxymethyl)propane-1,3-diyl bis(1H-pyrrole-2-carboxylate)

The title compound, C<sub>15</sub>H<sub>18</sub>N<sub>2</sub>O<sub>6</sub>, has two pyrrole–carboxylate groups which show different conformations. It forms tapes via N–H···O and O–H···O hydrogen bonds. The tapes are connected through pairs of N–H···O hydrogen bonds.

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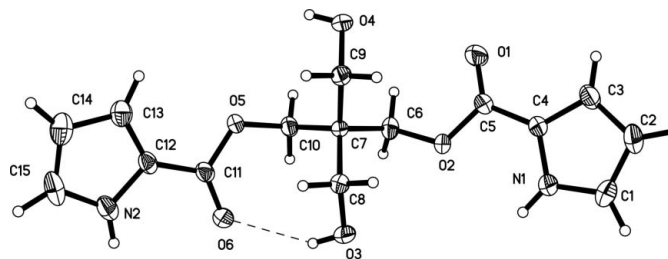
Comment

Pyrrole-based compounds have frequently been observed as hosts for neutral molecules (Fang *et al.*, 2004) and anionic species (Yin *et al.*, 2004). It was found that these compounds also have the ability to form higher-order self-assembled ensembles and aggregates in the solid state via hydrogen bonds (Sessler *et al.*, 2003). Here, we report the self-assembly of the title compound, (I), via conventional N–H···O and O–H···O hydrogen bonds.

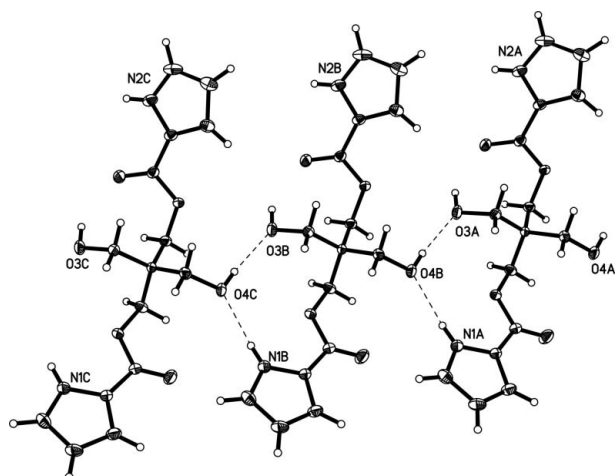


Treatment of 2-trichloroacetylpyrrole with pentaerythritol and triethylamine in refluxed acetonitrile solution gave the title compound directly. The molecular structure of (I) is shown in Fig. 1.

In the solid state, the two pyrrole-2-carboxylate groups have different conformations. One is in a *syn* conformation, with the carbonyl group arranged *syn* to its adjacent pyrrole NH



**Figure 1**  
 The structure and atomic numbering scheme of (I), drawn with 30% probability displacement ellipsoids. The dashed line indicates a hydrogen bond.

**Figure 2**

The tape-like self-assembly of molecules of (I) via O—H...O and N—H...O hydrogen bonds (dashed lines). [Symmetry codes: (A)  $x - 1, y, z$ ; (B)  $x, y, z$ ; (C)  $x + 1, y, z$ .]

group, and the other is in an *anti* conformation, with the carbonyl group arranged *anti* to its adjacent pyrrole NH group.

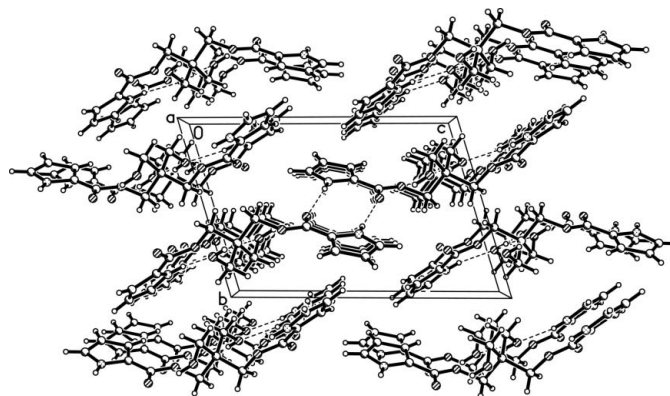
There is an intramolecular O—H...O hydrogen bond between the hydroxyl group (O3—H) and the carbonyl group (O6=C11) of the *syn* pyrrole-2-carboxylate group (Table 2). Molecules of (I) assemble as a tape through N—H...O and O—H...O hydrogen bonds, as shown in Fig. 2. The tapes are further linked by a pair of N—H...O hydrogen bonds between neighbouring *syn* pyrrole-2-carboxylate groups (Fig. 3).

## Experimental

2-Trichloroacetylpyrrole (527 mg, 2.5 mmol), pentaerythritol (136 mg, 1 mmol) and triethylamine (0.5 ml) were added to acetonitrile (20 ml), and the mixture was refluxed for 12 h. The solution was evaporated under reduced pressure and the residue was purified by column chromatography on silica gel with ethyl acetate–petroleum ether (2:1 *v/v*), affording the title compound (177 mg, 55%). Spectroscopic analysis:  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ ,  $\delta$ , p.p.m.): 9.20 (*b*, 2H, NH), 6.99 (*s*, 2H, —CH—), 6.96 (*s*, 2H, —CH—), 6.29 (*s*, 2H, —CH—), 4.40 (*s*, 4H, —CH<sub>2</sub>—O), 3.66 (*d*, 4H,  $J = 4.8$  Hz, —CH<sub>2</sub>—OH). ESI-MS (*m/z*): 322.9 ( $M+\text{H}^+$ ). Analysis calculated for  $\text{C}_{15}\text{H}_{18}\text{N}_2\text{O}_6$ : C 55.90, H 5.63, N 8.69%; found: C 55.67, H 5.64, N 8.66%.

### Crystal data

$\text{C}_{15}\text{H}_{18}\text{N}_2\text{O}_6$   $Z = 2$   
 $M_r = 322.31$   $D_x = 1.380 \text{ Mg m}^{-3}$   
 Triclinic,  $P\bar{1}$  Mo  $K\alpha$  radiation  
 Cell parameters from 1172 reflections  
 $a = 6.434$  (2) Å  $\theta = 2.3\text{--}24.7^\circ$   
 $b = 9.212$  (3) Å  $\mu = 0.11 \text{ mm}^{-1}$   
 $c = 13.715$  (4) Å  $T = 294$  (2) K  
 $\alpha = 73.202$  (5)° Block, colourless  
 $\beta = 85.533$  (5)°  $0.32 \times 0.20 \times 0.10 \text{ mm}$   
 $\gamma = 89.330$  (5)°

**Figure 3**

A crystal packing diagram, showing tapes held together via pairs of N—H...O hydrogen bonds (dashed lines).

### Data collection

Bruker SMART CCD area-detector diffractometer 2713 independent reflections  
 1733 reflections with  $I > 2\sigma(I)$   
 $\varphi$  and  $\omega$  scans  $R_{\text{int}} = 0.021$   
 Absorption correction: multi-scan ( $\text{SADABS}$ ; Bruker, 1997)  $\theta_{\text{max}} = 25.0^\circ$   
 $T_{\text{min}} = 0.949$ ,  $T_{\text{max}} = 0.989$   $h = -7 \rightarrow 6$   
 3971 measured reflections  $k = -10 \rightarrow 10$   
 $l = -16 \rightarrow 16$

### Refinement

Refinement on  $F^2$   $w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 0.0475P]$   
 $R[F^2 > 2\sigma(F^2)] = 0.041$  where  $P = (F_o^2 + 2F_c^2)/3$   
 $wR(F^2) = 0.099$   $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $S = 1.02$   $\Delta\rho_{\text{max}} = 0.15 \text{ e \AA}^{-3}$   
 2713 reflections  $\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$   
 222 parameters  
 H atoms treated by a mixture of independent and constrained refinement

**Table 1**

Selected geometric parameters (Å, °).

O1—C5	1.208 (2)	N1—C1	1.346 (3)
O2—C5	1.346 (2)	N1—C4	1.371 (3)
O2—C6	1.442 (2)	N1—H1	0.82 (2)
O3—H3	0.82 (3)	N2—C15	1.347 (3)
O5—C11	1.341 (2)	N2—C12	1.373 (3)
O5—C10	1.445 (2)	N2—H2	0.86 (2)
O6—C11	1.225 (2)		
C5—O2—C6	118.39 (16)	N1—C4—C5	123.65 (18)
C8—O3—H3	107 (2)	O1—C5—O2	122.98 (19)
C9—O4—H4	107.1 (17)	O1—C5—C4	124.73 (19)
C11—O5—C10	117.84 (16)	O2—C5—C4	112.29 (18)
C1—N1—C4	109.05 (19)	O6—C11—O5	123.1 (2)
C1—N1—H1	128.4 (15)	O6—C11—C12	125.29 (19)
C4—N1—H1	122.5 (15)	O5—C11—C12	111.61 (19)
C15—N2—C12	108.7 (2)	C13—C12—N2	107.6 (2)
C15—N2—H2	128.8 (18)	C13—C12—C11	132.0 (2)
C12—N2—H2	122.2 (18)	N2—C12—C11	120.4 (2)
N1—C4—C3	107.05 (19)		
C6—O2—C5—C4	−176.00 (16)	C10—O5—C11—C12	−176.30 (16)
N1—C4—C5—O1	−170.5 (2)	O6—C11—C12—C13	173.9 (2)
C3—C4—C5—O1	7.4 (4)	O5—C11—C12—C13	−5.4 (3)
N1—C4—C5—O2	9.2 (3)	O6—C11—C12—N2	−3.7 (3)
C3—C4—C5—O2	−172.9 (2)	O5—C11—C12—N2	176.99 (17)

**Table 2**  
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3 $\cdots$ O6	0.82 (3)	2.20 (3)	2.937 (2)	150 (3)
O4—H4 $\cdots$ O3 <sup>i</sup>	0.87 (3)	1.92 (3)	2.769 (2)	166 (2)
N1—H1 $\cdots$ O4 <sup>ii</sup>	0.82 (2)	2.17 (2)	2.989 (3)	173 (2)
N2—H2 $\cdots$ O6 <sup>iii</sup>	0.86 (2)	2.12 (3)	2.936 (3)	160 (2)

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

H atoms bound to O and N were located in a difference map and refined freely. Other H atoms were placed in difference Fourier map and positioned geometrically, with  $C-H = 0.93$  or  $0.97$  Å, and included in the final cycles of refinement using a riding model, with  $U_{iso}(H) = 1.2U_{eq}(C)$ .

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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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