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Ethyl 5-cyano-1*H*-pyrrole-2-carboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.053
wR factor = 0.159
Data-to-parameter ratio = 12.4

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

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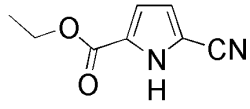
The title compound, $C_8H_8N_2O_2$, was prepared by dehydration of a pyrroleoxime in the presence of triphosgene. The molecules self-assemble into a layered structure through hydrogen bonding. All non-H atoms lie on a crystallographic mirror plane.

Comment

Hydrogen bonding in organic crystals has been established as a reliable force for organic crystal engineering. Pyrrole derivatives have recently shown high potential in effective self-assembly involving hydrogen bonding. Here we report the self-assembly of ethyl 5-cyano-1*H*-pyrrole-2-carboxylate, (I), *via* conventional N···H—N hydrogen bonds and non-conventional C—H···O hydrogen bonds.



(I)

The title compound was synthesized by dehydration of the oxime precursor in the presence of triphosgene. The molecular structure is shown in Fig. 1. The whole molecule is planar, except for ethyl H atoms. The carbonyl C═O is arranged *syn* to the pyrrole NH group. Molecules assemble as a tape through zigzag N···H—N hydrogen bonds assisted by pyrrole C—H···O═C hydrogen bonds. The tapes are linked by hydrophobic interactions of the ethyl groups and form a layer structure (Fig. 2). The distance between layers is half the *b*-axis repeat, 3.355 (2) Å.

Experimental

Ethyl 5-[(hydroxyimino)methyl]-1*H*-pyrrole-2-carboxylate (0.368 g, 2 mmol) was dissolved in CH_2Cl_2 (10 ml) and triethylamine (0.5 ml) was added. A solution of triphosgene (0.594 g, 2 mmol) in CH_2Cl_2 (5 ml) was added dropwise to this solution in an ice bath and the resulting solution was kept for 24 h at room temperature. The solu-

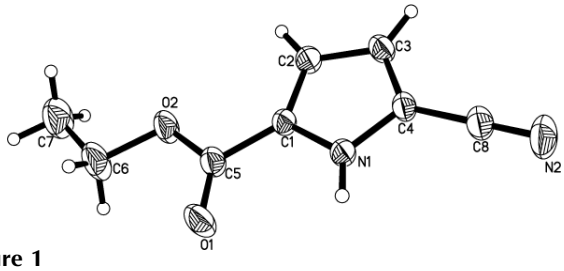


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids.

tion was washed twice with water and purified by flash chromatography to give the title compound as colorless crystals. Yield 55%, m.p. 392–393 K, ^{1}H NMR (300 MHz, DMSO-d_6, p.p.m.), 1.40 (*t*, 3H, *J* = 7 Hz, CH_3), 4.42 (*q*, 2H, *J* = 7 Hz, CH_2), 6.83 (*s*, 1H, CH), 6.90 (*s*, 1H, CH), 10.66 (*s*, 1H, NH); IR (KBr disk, cm^{-1}), 3244, 3112, 2237, 1704, 1321, 1248. 20 mg of (I) was dissolved in 15 ml dioxane; the solution was kept at room temperature for 15 d and natural evaporation gave colorless single crystals of (I) suitable for X-ray analysis.

Crystal data

$C_8H_8N_2O_2$
M_r = 164.16
Orthorhombic, *Pnma*
a = 7.866 (3) Å
b = 6.710 (3) Å
c = 15.959 (7) Å
V = 842.3 (6) Å^3
Z = 4
D_x = 1.295 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 762 reflections
θ = 2.6–24.5°
μ = 0.10 mm^{-1}
T = 293 (2) K
Block, colorless
0.32 × 0.28 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
2209 measured reflections
921 independent reflections
664 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 24.5°
$h = -5 \rightarrow 9$
$k = -8 \rightarrow 4$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.159
S = 1.12
921 reflections
74 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

H atoms were positioned geometrically, with C—H = 0.93–0.98 Å, and refined as riding, with U_{iso}(H) = 1.2U_{eq}(carrier).

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

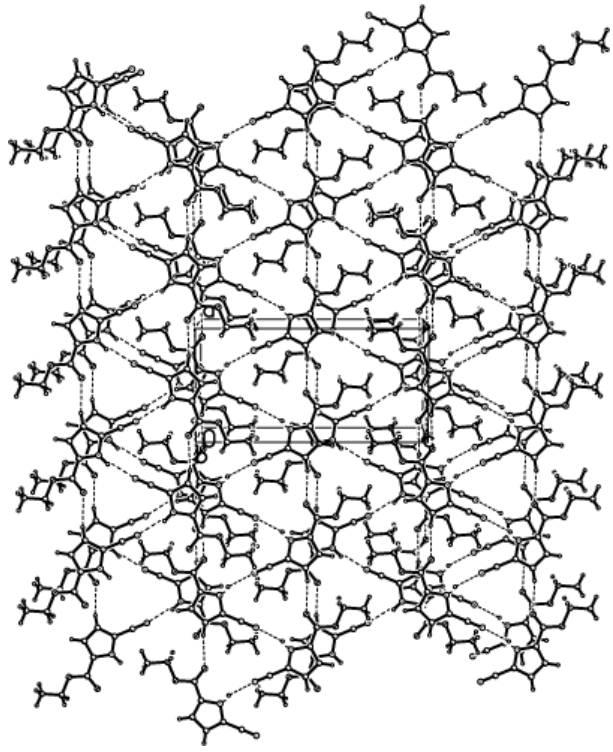


Figure 2
The crystal structure of (I), viewed along the *b* axis

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References

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