Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-Picolinoyl-3-[(2-pyridyl)(2,2,2-trifluoroacetamido)methyl]urea

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.005 Å
R factor = 0.058
wR factor = 0.177
Data-to-parameter ratio = 11.8

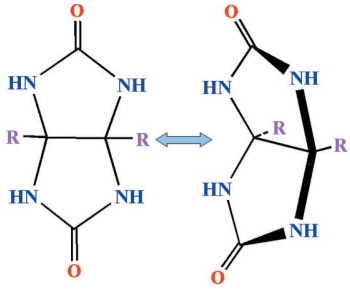
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 16 November 2006
Accepted 13 December 2006

The title compound, $C_{15}H_{12}F_3N_5O_3$, is a by-product isolated from the preparation of di-2-pyridiylglycoluril. The molecule contains two classical intramolecular N—H···N and N—H···O hydrogen bonds, and its crystal structure is stabilized mostly by intermolecular N—H···O hydrogen bonds.

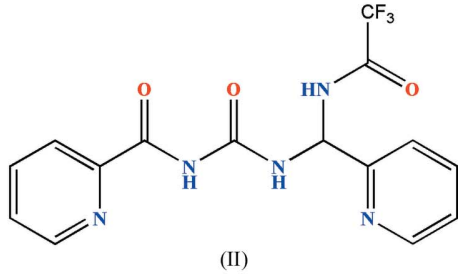
Comment

The glycoluril skeleton, (I), is an important building block for the preparation of a wide variety of supramolecular assemblies, including cucurbit[*n*]uril homologues (*n* = 5, 7, 8 and 10) and their derivatives (Lee *et al.*, 2003), molecular clips and baskets (Rowan *et al.*, 1999), and molecular capsules (Hof *et al.*, 2002). The title compound, (II), is obtained as a by-product in the preparation of di-2-pyridinylglycoluril.



R= -H, -CH_3, -COOEt, -Ph, ...

(I)



(II)

The molecular structure of (II) is shown in Fig. 1. The two pyridyl rings are almost coplanar [dihedral angle 5.4 (3)°]. Classical intramolecular N—H···N and N—H···O hydrogen bonds (Table 1, entries 1 and 2) may account for the observed molecular conformation. The infinite one-dimensional chain packing structure is stabilized mostly by intermolecular N—H···O hydrogen bonds (Table 1, entry 3).

Experimental

Urea (6.0 g, 0.1 mol), 1,2-di(pyridin-2-yl)ethane-1,2-dione (10.6 g, 0.05 mol), trifluoroacetic acid (10 ml) and benzene (200 ml) were refluxed at 400 K, until water production ceased (5–6 h). After being filtered off and washed with ethanol, the crude product was dissolved in ethanol and stirred overnight at 298 K. The final expected glycoluril derivative was obtained (7.0–8.0 g, 47–54% yield), together with (II) as a by-product, isolated from the filtrate. Crystals of (II) suitable for data collection were obtained by slow evaporation of a chloroform–methanol solution (5:1 *v*/*v*) at 298 K.

Crystal data

$C_{15}H_{12}F_3N_5O_3$
M_r = 367.30
Triclinic, $P\overline{1}$
a = 4.9095 (12) Å
b = 12.754 (3) Å
c = 13.072 (3) Å
α = 85.969 (4)°
β = 79.721 (4)°
γ = 86.585 (4)°
V = 802.4 (3) Å^3
Z = 2
D_x = 1.520 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 292 (2) K
Block, colourless
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART 4K area-detector diffractometer
φ and ω scans
Absorption correction: none
3937 measured reflections
2775 independent reflections
1725 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.177
S = 1.03
2775 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0912P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N1	0.86	2.19	2.615 (4)	111
N3—H3A···O1	0.86	2.16	2.781 (3)	129
N4—H4A···O2^{i}	0.86	2.02	2.808 (3)	151

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

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 Å (aromatic CH), C—H = 0.98 Å (methine CH) or N—H = 0.86 Å (amine NH), and with U_{iso}(H) = 1.2U_{eq}(C,N).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 1997).

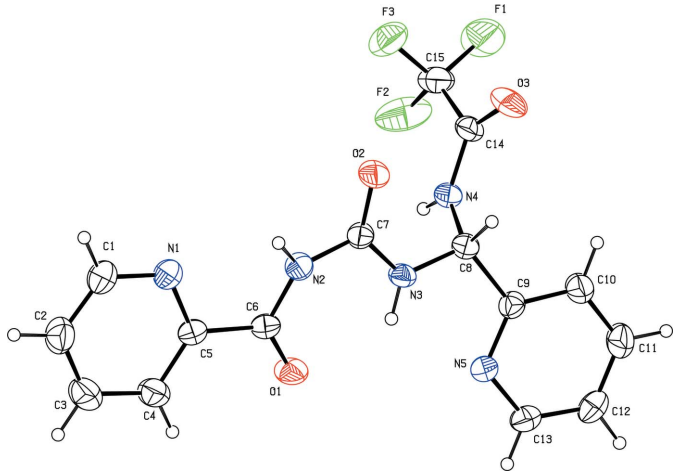


Figure 1
The molecular structure of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

The authors are grateful to Central China Normal University, and to Dr Xiang-Gao Meng for the X-ray data collection.

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