

James L. Wardell,^a John N. Low^b
 and Christopher Glidewell^{c*}

^aInstituto de Química, Departamento de Química Inorgânica, Universidade Federal do Rio de Janeiro, CP 68563, 21945-970 Rio de Janeiro, RJ, Brazil, ^bDepartment of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and ^cSchool of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland

Correspondence e-mail: cg@st-andrews.ac.uk

Key indicators

Single-crystal X-ray study
 T = 120 K
 Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
 R factor = 0.040
 wR factor = 0.094
 Data-to-parameter ratio = 15.0

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

5-Amino-1-(2-chloronicotinoyl)-3-trifluoromethyl-1*H*-1,2,4-triazole: hydrogen-bonded sheets of alternating $R_2^2(8)$ and $R_6^6(36)$ rings

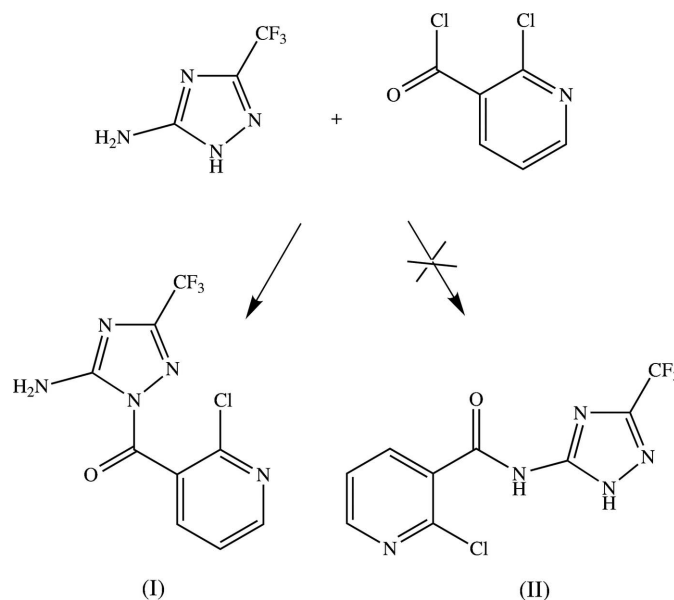
The molecules of the title compound, $\text{C}_9\text{H}_5\text{ClF}_3\text{N}_5\text{O}$, are linked by two independent $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds into sheets containing alternating $R_2^2(8)$ and $R_6^6(36)$ rings.

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Comment

We have recently reported the molecular and supramolecular structures of a number of *N*-aryl-2-chloronicotinamides obtained from the reactions between 2-chloronicotinoyl chloride and substituted anilines (de Souza *et al.*, 2005; Cuffini *et al.*, 2006). In a continuation of this study, we now report the structure of the title compound, (I), obtained from the reaction between 2-chloronicotinoyl chloride and 5-amino-3-trifluoromethyl-1*H*-1,2,4-triazole. The formation of (I) was unexpected, as reaction at the exocyclic amino group was expected to yield the isomeric compound, (II) (see scheme).



The carbonyl group of (I) is almost coplanar with the triazole ring (Fig. 1, Table 1) and this is possibly controlled by the intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond (Table 2). On the other hand, the pyridyl ring is rotated significantly out of this plane. The bond distances in the triazole ring provide evidence for strong bond fixation within this ring.

The molecules of compound (I) are linked by two independent $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds (Table 2) into sheets, whose formation can readily be analysed in terms of two simple substructures, each utilizing just one hydrogen bond. One substructure is finite and zero-dimensional, while the other is one-dimensional.

Experimental

A mixture of 2-chloronicotinoyl chloride (0.88 g, 5 mmol) and 5-amino-3-trifluoromethyl-1*H*-1,2,4-triazole (0.76 g, 5 mmol) (Lopyrev & Rakhmatulina, 1983) in 1,2-dichloroethane (15 ml) was heated under reflux for 1 h. The mixture was then cooled and the solvent removed under reduced pressure. The resulting solid product, (I), was recrystallized from ethyl acetate to give crystals suitable for single-crystal X-ray diffraction.

Crystal data

$C_9H_5ClF_3N_5O$
 $M_r = 291.63$
 Monoclinic, $P2_1/n$
 $a = 4.64770$ (10) Å
 $b = 19.7414$ (10) Å
 $c = 12.3721$ (5) Å
 $\beta = 91.147$ (3)°
 $V = 1134.94$ (8) Å³

$Z = 4$
 $D_x = 1.707$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.38$ mm⁻¹
 $T = 120$ (2) K
 Needle, colourless
 $0.26 \times 0.06 \times 0.05$ mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
 $T_{\min} = 0.927$, $T_{\max} = 0.981$

14659 measured reflections
 2588 independent reflections
 1923 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$
 $\theta_{\text{max}} = 27.6^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.094$
 $S = 1.04$
 2588 reflections
 172 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2 + 0.619P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.25$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

N1–N2	1.391 (2)	N1–C27	1.401 (2)
N2–C3	1.305 (2)	C27–O27	1.209 (2)
C3–N4	1.364 (2)	C3–C31	1.488 (3)
N4–C5	1.328 (2)	C5–N5	1.324 (2)
C5–N1	1.392 (2)		
N2–N1–C27–O27	−178.39 (17)	N1–C27–C23–C22	70.4 (2)
N2–N1–C27–C23	0.1 (3)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N5–H5A \cdots O27	0.88	2.25	2.836 (2)	124
N5–H5A \cdots N21 ⁱ	0.88	2.40	3.053 (2)	131
N5–H5B \cdots N4 ⁱⁱ	0.88	2.13	2.985 (2)	163

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$; (ii) $-x + 1, -y + 1, -z + 1$.

All H atoms were located in difference maps and then treated as riding atoms, with C–H = 0.95 Å and N–H = 0.88 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$.

Data collection: COLLECT (Nonius, 1999); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduction: DENZO and COLLECT; program(s) used to solve structure: OSCAIL (McArdle, 2003) and SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: OSCAIL and SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97 and PRPKAPPA (Ferguson, 1999).

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