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N-(5-Amino-1*H*-1,2,4-triazol-3-yl)-pyridine-2-carboxamide

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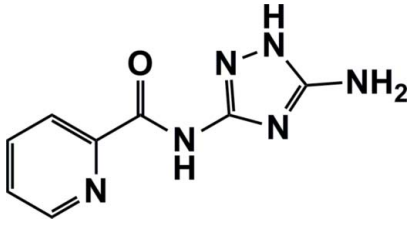
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.107; data-to-parameter ratio = 10.3.

The title compound, $C_8H_8N_6O$, was obtained by the reaction of 3,5-diamino-1,2,4-triazole with ethyl 2-picolinate in a glass oven. The dihedral angles formed between the plane of the amide group and the pyridine and triazole rings are 11.8 (3) and 5.8 (3)°, respectively. In the crystal, an extensive system of classical N—H···N and N—H···O hydrogen bonds generate an infinite three-dimensional network.

Related literature

For background to triazole derivatives, see: Aromí *et al.* (2011); Olguín *et al.* (2012). For related triazole structures, see: Allouch *et al.* (2008); Ouakkaf *et al.* (2011). For structures of metal complexes with related triazoles, see: Ferrer *et al.* (2004, 2012). For the synthesis of triazoles, see: Chernyshev *et al.* (2005). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_8H_8N_6O$
M_r = 204.20
Tetragonal, $P4_12_12$
a = 9.5480 (5) Å
c = 21.9570 (9) Å
V = 2001.69 (17) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.15 × 0.09 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
4484 measured reflections
1407 independent reflections
915 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.107
S = 1.07
1407 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N21—H21···N23^{i}	0.86	2.02	2.788 (3)	149
N21—H21···O17^{i}	0.86	2.41	3.061 (3)	133
N18—H18···N20^{ii}	0.86	2.45	3.253 (3)	155
N22—H22*A*···O17^{i}	0.86	2.08	2.860 (3)	150
N22—H22*B*···N20^{iii}	0.86	2.26	3.068 (3)	157

Symmetry codes: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{4}$; (ii) $-y, -x, -z+\frac{1}{2}$; (iii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{4}$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *DIAMOND* (Brandenburg & Putz, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2547).

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supporting information

Acta Cryst. (2013). E**69**, o227–o228 [doi:10.1107/S1600536813000123]

N-(5-Amino-1*H*-1,2,4-triazol-3-yl)pyridine-2-carboxamide

Javier Hernández-Gil, Sacramento Ferrer, Rafael Ballesteros and Alfonso Castiñeiras

S1. Comment

A significantly large variety of 1,2,4-triazole-based compounds have been prepared to serve as ligands with the aim of obtaining discrete polynuclear metal complexes or polymeric coordination networks, owing to the ability of the 1,2,4-triazole ring to bridge metal ions through different coordination ways (Aromí *et al.*, 2011; Olguín *et al.*, 2012). Usually the 1,2,4-triazole-based family of ligands are classified in three categories (Aromí *et al.*, 2011): (*a*) those containing an unique coordinative ring, (*b*) those possessing two or more coordinative rings linked by a spacer, and (*c*) the *mixed* ligands, which present two or more functional groups. Most of the 3,5-disubstituted derivatives can be included in the last category (Allouch *et al.*, 2008; Ouakkaf *et al.*, 2011).

Our group has been reporting on the synthesis and structure of some 3,5-disubstituted triazole-based ligands, *i.e.* 5-amino-3-pyridin-2-yl-1,2,4-triazole (Ferrer *et al.*, 2004) and 3-acetylamino-5-amino-1,2,4-triazole (Ferrer *et al.*, 2012). In those cases, single crystals of the ligands suitable for X-ray analysis were not obtained. Instead, crystal structures of some of their Cu^{II} complexes could be determined, thus confirming the structure of the triazole, either in neutral or in anionic form. In this work we describe a novel compound of this series, namely: 5-amino-3-picolinamido-1*H*-1,2,4-triazole or 5-amino-3-(pyridin-2-yl-acetamido)-1*H*-1,2,4-triazole (abbreviated as H_2V to account for the presence of two acidic H atoms), for which it has been possible to solve the crystal structure.

The obtained H_2V species is an attractive ligand since it presents 5 to 7 donor atoms (depending on the degree of deprotonation) but also the possibility of forming different chelating rings when coordinated to metals. Besides, in metal complexes the pyridyl ring often rotates around the single C–C bond leading to different binding conformations (Ouakkaf *et al.*, 2011). This enlarges its capability to produce novel metal-organic structures.

As shown in Figure 1, the NH hydrogen is *trans* to the C=O group, as is observed for all N monosustituted amides. Molecular dimensions, such as the C=O bond length of 1.227 (3) Å and the central C–N–C amide angle of 127.40 (17)°, may be considered normal.

In the crystal packing, the triazole ligands are linked by pairs of weak N—H···N hydrogen bonds involving the H18 and N20 atoms, thus generating a characteristic $R^2_2(8)$ ring motif (Bernstein *et al.*, 1995) (Fig. 2). Moreover, the molecules are also linked by N—H···N and N—H···O hydrogen bonds, forming fused non-centrosymmetric rings $R^2_2(7)$, $R^2_1(6)$ and $R^1_2(6)$ and giving rise to one-dimensional tapes parallel to the [010] and [100] directions (Fig. 3). These tapes joined by the $R_2^2(8)$ motif of N-H···N hydrogen bonds form a three dimensional framework (Fig.4).

S2. Experimental

An evaporating flask containing 3,5-diamino-1,2,4-triazole (41.4 mmol, 4.10 g) and ethyl 2-picolinate (6.3 ml, 7 g, 46.3 mmol) was connected to a glass oven and the reaction temperature was slowly raised to 210 °C. The mixture was stirred (rotated) for 4 h. At this point, a vacuum pump was connected during 60 minutes to remove the excess of ethyl 2-picolinate. Afterwards, the reaction was cooled down to room temperature and the mixture solidified. The crude product

was washed with ethanol and acetone and then recrystallized from methanol to give analytically pure crystals.

S3. Refinement

All H atoms were positioned geometrically and were treated as riding on their parent atoms, with C—H distances of 0.93 Å and N—H distances of 0.86 Å with U_{iso}(H) = 1.2U_{eq}(C/N). In the absence of significant anomalous dispersion, Friedel pairs were merged.

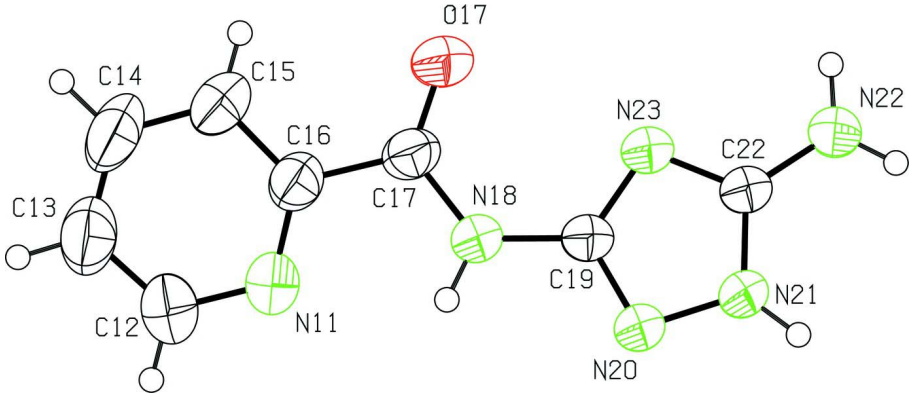

Figure 1

Molecular structure of the title molecule with atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

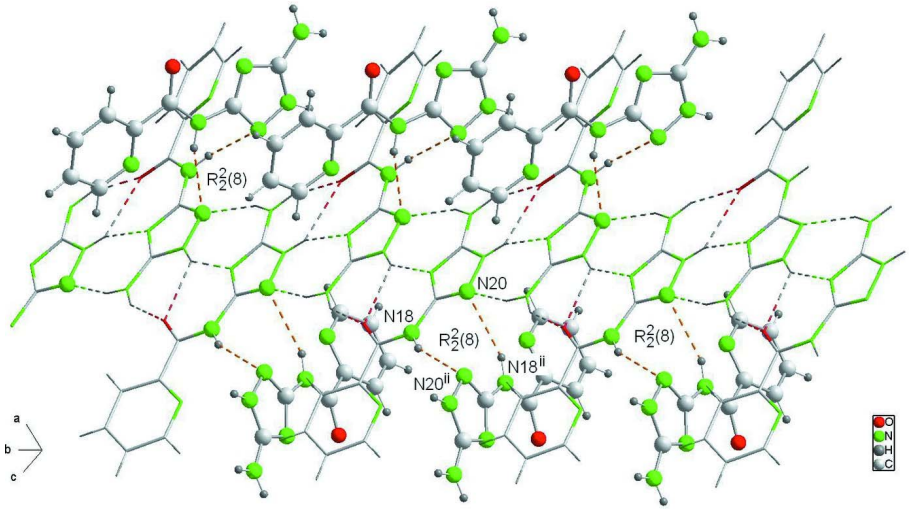

Figure 2

Scheme with details of the *crossing* of two chains of molecules (along the *cb* plane). Hydrogen bonds are shown as dashed lines. Symmetry code: (ii) -*y*, -*x*, -*z* + 1/2

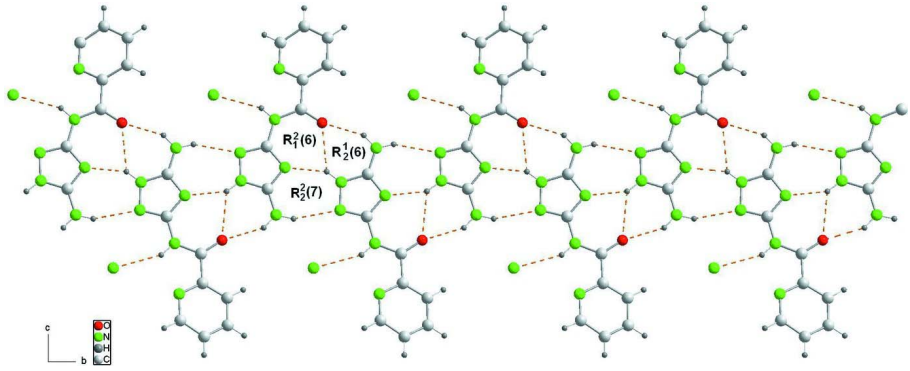

Figure 3

Tapes of title molecules via N—H···N and N—H···O interactions seen along the [100] direction. Hydrogen bonds are shown as orange dashed lines.

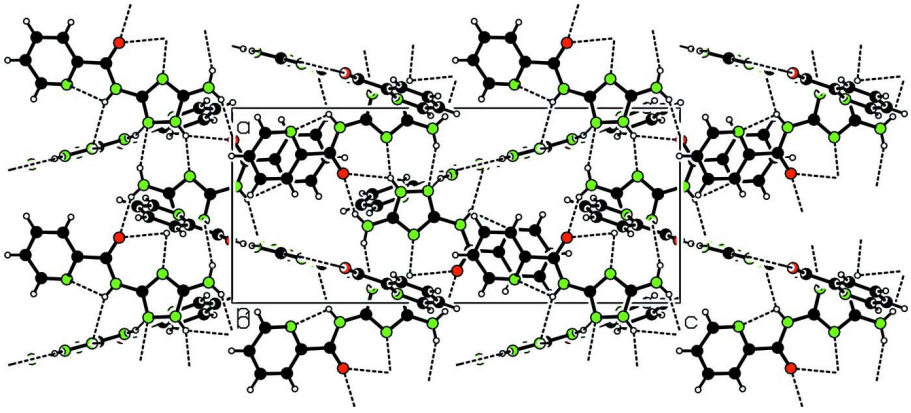

Figure 4

A view of the unit-cell content of the title compound in projection down the *b* axis. Hydrogen bonds are shown as dashed lines.

N-(5-Amino-1*H*-1,2,4-triazol-3-yl)pyridine-2-carboxamide

Crystal data

$C_8H_8N_6O$
M_r = 204.20
Tetragonal, $P4_12_12$
Hall symbol: P 4abw 2nw
a = 9.5480 (5) Å
c = 21.9570 (9) Å
V = 2001.69 (17) Å^3
Z = 8
F(000) = 848
D_x = 1.355 Mg m^{-3}
Melting point: 494(1) K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2353 reflections
θ = 1.0–27.5°
μ = 0.10 mm^{-1}
T = 293 K
Prism, colourless
0.15 × 0.09 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 9 pixels mm^{-1}
ω and phi scans
4484 measured reflections
1407 independent reflections
915 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

θ_{max} = 27.5°, θ_{min} = 2.3°
h = −12→12
k = −8→8
l = −28→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.107
S = 1.07
1407 reflections
137 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0557P)^2 + 0.0085P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.013 (2)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.
Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
O17	0.1793 (2)	0.33529 (18)	0.25244 (8)	0.0726 (6)
N11	0.0462 (3)	0.1210 (3)	0.36912 (9)	0.0708 (7)
N18	0.1596 (2)	0.09855 (19)	0.25946 (8)	0.0513 (5)
H18	0.1399	0.0318	0.2843	0.062*
N20	0.2076 (2)	−0.0746 (2)	0.18712 (8)	0.0495 (5)
N21	0.2450 (2)	−0.0695 (2)	0.12653 (8)	0.0503 (5)
H21	0.2593	−0.1412	0.1036	0.060*
N22	0.2887 (3)	0.1049 (2)	0.05253 (9)	0.0797 (9)
H22A	0.3046	0.0438	0.0246	0.096*
H22B	0.2939	0.1928	0.0442	0.096*
N23	0.2279 (2)	0.14920 (19)	0.15549 (8)	0.0543 (6)
C12	−0.0006 (4)	0.1273 (4)	0.42633 (13)	0.0885 (11)
H12	−0.0387	0.0467	0.4434	0.106*
C13	0.0043 (4)	0.2465 (4)	0.46156 (14)	0.0871 (10)
H13	−0.0288	0.2460	0.5014	0.104*
C14	0.0583 (5)	0.3637 (4)	0.43690 (15)	0.1011 (12)
H14	0.0627	0.4458	0.4596	0.121*
C15	0.1072 (4)	0.3613 (3)	0.37768 (13)	0.0877 (11)
H15	0.1451	0.4412	0.3599	0.105*
C16	0.0983 (3)	0.2377 (3)	0.34567 (11)	0.0585 (7)
C17	0.1488 (3)	0.2302 (3)	0.28160 (11)	0.0541 (6)

C19	0.1992 (2)	0.0590 (2)	0.20093 (10)	0.0463 (6)
C22	0.2557 (3)	0.0633 (2)	0.10904 (10)	0.0503 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O17	0.1132 (17)	0.0408 (10)	0.0639 (11)	−0.0008 (10)	0.0054 (11)	0.0032 (9)
N11	0.0937 (19)	0.0633 (16)	0.0552 (13)	−0.0053 (13)	0.0148 (12)	−0.0079 (11)
N18	0.0701 (14)	0.0370 (11)	0.0468 (11)	−0.0015 (10)	0.0061 (10)	−0.0016 (9)
N20	0.0677 (14)	0.0356 (11)	0.0453 (10)	0.0003 (9)	0.0062 (9)	−0.0003 (9)
N21	0.0723 (14)	0.0345 (11)	0.0442 (10)	0.0013 (10)	0.0061 (9)	−0.0020 (8)
N22	0.147 (3)	0.0395 (13)	0.0522 (13)	0.0045 (14)	0.0264 (15)	0.0046 (10)
N23	0.0790 (15)	0.0351 (10)	0.0487 (11)	−0.0009 (10)	0.0068 (11)	0.0014 (9)
C12	0.115 (3)	0.085 (2)	0.0647 (18)	−0.010 (2)	0.0275 (18)	−0.0075 (17)
C13	0.104 (3)	0.093 (3)	0.0643 (18)	0.014 (2)	0.0149 (17)	−0.0163 (19)
C14	0.148 (4)	0.080 (3)	0.076 (2)	0.018 (3)	0.009 (2)	−0.033 (2)
C15	0.136 (3)	0.0541 (19)	0.073 (2)	0.0064 (19)	0.0134 (19)	−0.0150 (16)
C16	0.0715 (18)	0.0512 (17)	0.0528 (14)	0.0080 (13)	0.0001 (12)	−0.0059 (13)
C17	0.0664 (16)	0.0402 (14)	0.0558 (14)	0.0025 (12)	−0.0052 (12)	−0.0005 (12)
C19	0.0576 (15)	0.0364 (13)	0.0448 (13)	−0.0009 (11)	0.0011 (11)	0.0022 (10)
C22	0.0686 (17)	0.0355 (13)	0.0468 (13)	−0.0007 (12)	0.0043 (12)	−0.0005 (11)

Geometric parameters (Å, º)

O17—C17	1.225 (3)	N22—H22B	0.8600
N11—C16	1.324 (3)	N23—C22	1.335 (3)
N11—C12	1.335 (3)	N23—C19	1.347 (3)
N18—C17	1.352 (3)	C12—C13	1.377 (4)
N18—C19	1.392 (3)	C12—H12	0.9300
N18—H18	0.8600	C13—C14	1.346 (5)
N20—C19	1.314 (3)	C13—H13	0.9300
N20—N21	1.378 (2)	C14—C15	1.382 (4)
N21—C22	1.329 (3)	C14—H14	0.9300
N21—H21	0.8600	C15—C16	1.376 (4)
N22—C22	1.340 (3)	C15—H15	0.9300
N22—H22A	0.8600	C16—C17	1.489 (3)
C16—N11—C12	117.0 (2)	C13—C14—C15	119.6 (3)
C17—N18—C19	127.3 (2)	C13—C14—H14	120.2
C17—N18—H18	116.4	C15—C14—H14	120.2
C19—N18—H18	116.3	C16—C15—C14	118.3 (3)
C19—N20—N21	101.78 (18)	C16—C15—H15	120.9
C22—N21—N20	109.41 (18)	C14—C15—H15	120.9
C22—N21—H21	125.3	N11—C16—C15	123.1 (2)
N20—N21—H21	125.3	N11—C16—C17	116.7 (2)
C22—N22—H22A	120.0	C15—C16—C17	120.2 (3)
C22—N22—H22B	120.0	O17—C17—N18	123.7 (2)
H22A—N22—H22B	120.0	O17—C17—C16	122.1 (2)

C22—N23—C19	102.32 (19)	N18—C17—C16	114.1 (2)
N11—C12—C13	123.7 (3)	N20—C19—N23	116.0 (2)
N11—C12—H12	118.1	N20—C19—N18	119.6 (2)
C13—C12—H12	118.1	N23—C19—N18	124.4 (2)
C14—C13—C12	118.3 (3)	N21—C22—N23	110.5 (2)
C14—C13—H13	120.8	N21—C22—N22	124.6 (2)
C12—C13—H13	120.8	N23—C22—N22	124.9 (2)
C19—N20—N21—C22	−0.1 (3)	N11—C16—C17—N18	−12.1 (4)
C16—N11—C12—C13	−0.8 (6)	C15—C16—C17—N18	167.8 (3)
N11—C12—C13—C14	0.6 (6)	N21—N20—C19—N23	−0.2 (3)
C12—C13—C14—C15	−0.2 (6)	N21—N20—C19—N18	178.5 (2)
C13—C14—C15—C16	0.2 (5)	C22—N23—C19—N20	0.5 (3)
C12—N11—C16—C15	0.8 (5)	C22—N23—C19—N18	−178.2 (2)
C12—N11—C16—C17	−179.3 (3)	C17—N18—C19—N20	178.4 (2)
C14—C15—C16—N11	−0.5 (5)	C17—N18—C19—N23	−3.0 (4)
C14—C15—C16—C17	179.6 (3)	N20—N21—C22—N23	0.4 (3)
C19—N18—C17—O17	−3.3 (4)	N20—N21—C22—N22	−179.1 (3)
C19—N18—C17—C16	177.7 (2)	C19—N23—C22—N21	−0.5 (3)
N11—C16—C17—O17	168.9 (3)	C19—N23—C22—N22	179.0 (3)
C15—C16—C17—O17	−11.3 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21—H21···N23^{i}	0.86	2.02	2.788 (3)	149
N21—H21···O17^{i}	0.86	2.41	3.061 (3)	133
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N22—H22*B*···N20^{iii}	0.86	2.26	3.068 (3)	157

Symmetry codes: (i) −*x*+1/2, *y*−1/2, −*z*+1/4; (ii) −*y*, −*x*, −*z*+1/2; (iii) −*x*+1/2, *y*+1/2, −*z*+1/4.