

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

N-(1,3-Thiazol-2-yl)-2-(2,4,6-trimethylphenyl)acetamide

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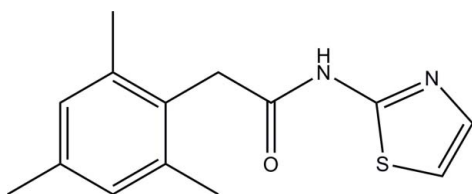
Received 8 July 2012; accepted 11 July 2012

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.040; wR factor = 0.103; data-to-parameter ratio = 22.6.

In the title compound, $\text{C}_{14}\text{H}_{16}\text{N}_2\text{OS}$, the thiazole ring is essentially planar (r.m.s. deviation = 0.005 Å) and it forms a dihedral angle of 75.21 (8)° with the benzene ring. In the crystal, molecules are linked into inversion dimers by pairs of $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds to generate $R_2^2(8)$ loops.

Related literature

For general background to and related structures of the title compound, see: Fun *et al.* (2011a,b, 2012). For graph-set notation of hydrogen bonds, see: Bernstein *et al.* (1995). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{16}\text{N}_2\text{OS}$	$V = 1283.1$ (3) Å ³
$M_r = 260.35$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 16.421$ (2) Å	$\mu = 0.24$ mm ⁻¹
$b = 4.6397$ (7) Å	$T = 100$ K
$c = 20.1144$ (18) Å	$0.30 \times 0.25 \times 0.09$ mm
$\beta = 123.150$ (7)°	

* Thomson Reuters ResearcherID: A-3561-2009.

§ Thomson Reuters ResearcherID: A-5525-2009.

Data collection

Bruker SMART APEXII DUO	13735 measured reflections
CCD diffractometer	3748 independent reflections
Absorption correction: multi-scan (SADABS; Bruker, 2009)	2999 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.932$, $T_{\max} = 0.979$	$R_{\text{int}} = 0.040$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$	166 parameters
$wR(F^2) = 0.103$	H-atom parameters constrained
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.39$ e Å ⁻³
3748 reflections	$\Delta\rho_{\text{min}} = -0.51$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H1}\cdots\text{N1}^i$	0.88	2.08	2.9623 (19)	175

 Symmetry code: (i) $-x + 2, -y + 1, -z + 2$.

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

The authors thank Universiti Sains Malaysia (USM) for a Research University Grant (No. 1001/PFIZIK/811160). BN also thanks UGC, New Delhi, and the Government of India for the purchase of chemicals through the SAP-DRS-Phase 1 programme.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6891).

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supporting information

Acta Cryst. (2012). E68, o2461 [https://doi.org/10.1107/S1600536812031595]

N*-(1,3-Thiazol-2-yl)-2-(2,4,6-trimethylphenyl)acetamide*Hoong-Kun Fun, Ching Kheng Quah, Prakash S. Nayak, B. Narayana and B. K. Sarojini****S1. Comment**

In continuation of our work on synthesis of amides (Fun *et al.*, 2011*a,b*, 2012), we report herein the crystal structure of the title compound.

In the title molecule (Fig. 1), the thiazol-2-yl ring (S1/N1/C1–C3) is essentially planar (r.m.s. deviation = 0.005 Å) and it forms a dihedral angle of 75.21 (8)° with the benzene ring (C6–C11). Bond lengths and angles are comparable to those in related structures (Fun *et al.*, 2011*a,b*, 2012).

In the crystal (Fig. 2), molecules are linked into an inversion dimer by pairs of intermolecular N2—H1···N1 hydrogen bonds (Table 1).

S2. Experimental

2,4,6-Trimethylphenylacetic acid (0.178 g, 1 mmol), 2-aminothiazole (0.1 g, 1 mmol) and 1-ethyl-3-(3-dimethylamino-propyl)-carbodiimide hydrochloride (1.0 g, 0.01 mol) were dissolved in dichloromethane (20 ml). The mixture was stirred in presence of triethylamine at 273 K for about 3 h. The contents were poured into 100 ml of ice-cold aqueous hydrochloric acid with stirring, which was extracted thrice with dichloromethane. Organic layer was washed with saturated NaHCO₃ solution and brine solution, dried and concentrated under reduced pressure to give the title compound. Colourless blocks were grown from an acetone and toluene (1:1) solvent mixture by the slow evaporation method (m.p. 457–459 K).

S3. Refinement

N-bound hydrogen atom was located in a difference Fourier map and refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{N})$ [N—H = 0.8794 Å]. The remaining H atoms were positioned geometrically and refined using a riding model with C—H = 0.95–0.99 Å and $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5 U_{\text{eq}}(\text{C})$. A rotating-group model was applied for the methyl groups.

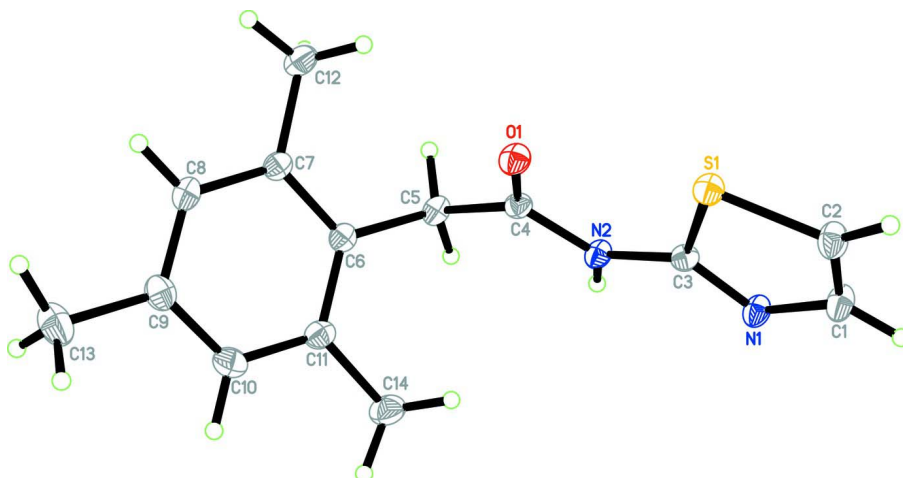


Figure 1

The molecular structure of the title compound showing 50% probability displacement ellipsoids for non-H atoms.

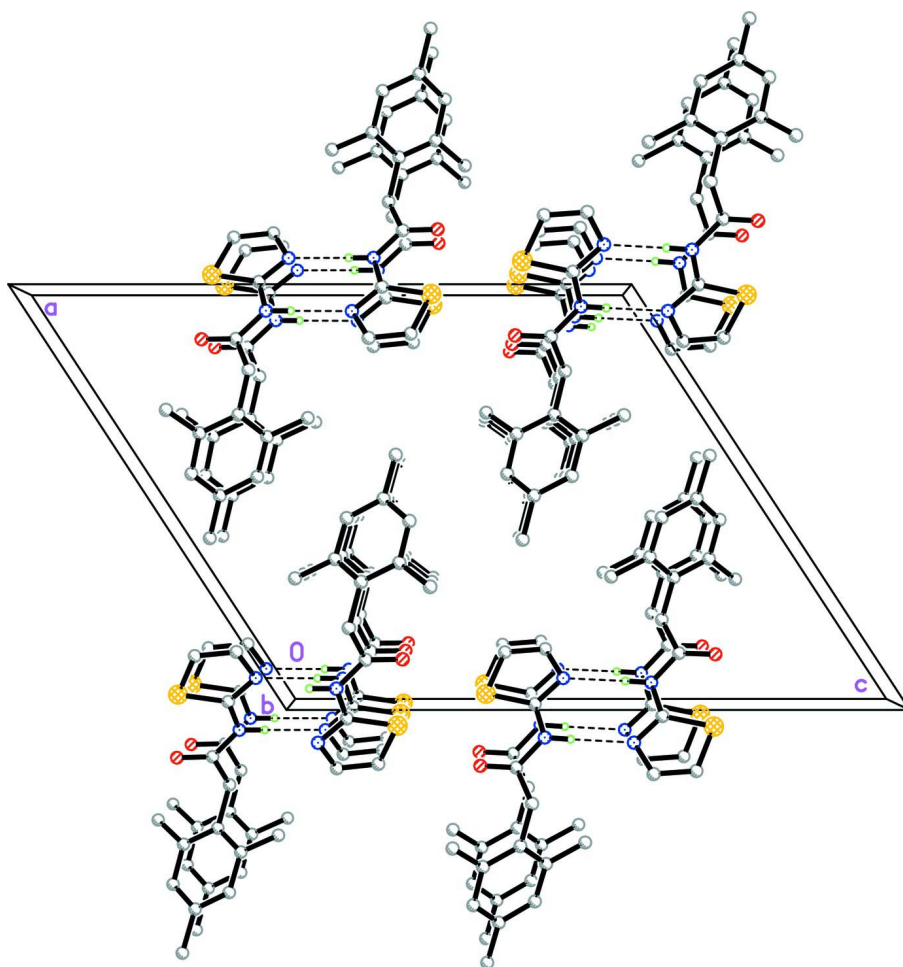


Figure 2

The crystal structure of the title compound, viewed along the *b* axis. H atoms not involved in hydrogen bonds (dashed lines) have been omitted for clarity.

N-(1,3-Thiazol-2-yl)-2-(2,4,6-trimethylphenyl)acetamide*Crystal data*C₁₄H₁₆N₂OS $M_r = 260.35$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 16.421 (2) \text{ \AA}$ $b = 4.6397 (7) \text{ \AA}$ $c = 20.1144 (18) \text{ \AA}$ $\beta = 123.150 (7)^\circ$ $V = 1283.1 (3) \text{ \AA}^3$ $Z = 4$ $F(000) = 552$ $D_x = 1.348 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4087 reflections

 $\theta = 3.0\text{--}29.9^\circ$ $\mu = 0.24 \text{ mm}^{-1}$ $T = 100 \text{ K}$

Block, colourless

 $0.30 \times 0.25 \times 0.09 \text{ mm}$ *Data collection*

Bruker SMART APEXII DUO CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2009)

 $T_{\min} = 0.932$, $T_{\max} = 0.979$

13735 measured reflections

3748 independent reflections

2999 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.040$ $\theta_{\max} = 30.1^\circ$, $\theta_{\min} = 1.5^\circ$ $h = -23 \rightarrow 20$ $k = -6 \rightarrow 6$ $l = -26 \rightarrow 28$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.040$ $wR(F^2) = 0.103$ $S = 1.03$

3748 reflections

166 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.7659P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.39 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.51 \text{ e \AA}^{-3}$ *Special details*

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	1.03056 (3)	0.78584 (8)	0.83439 (2)	0.01686 (10)
O1	0.86995 (7)	0.4661 (2)	0.75301 (6)	0.0180 (2)

N1	1.06903 (9)	0.7448 (3)	0.97704 (7)	0.0176 (2)
N2	0.93660 (8)	0.4568 (3)	0.88579 (7)	0.0151 (2)
H1	0.9347	0.3871	0.9256	0.018*
C1	1.13491 (11)	0.9354 (3)	0.97864 (9)	0.0206 (3)
H1A	1.1835	1.0281	1.0262	0.025*
C2	1.12628 (11)	0.9823 (3)	0.90878 (9)	0.0203 (3)
H2A	1.1671	1.1057	0.9017	0.024*
C3	1.01013 (10)	0.6518 (3)	0.90413 (8)	0.0145 (3)
C4	0.86909 (10)	0.3723 (3)	0.80944 (8)	0.0146 (3)
C5	0.79327 (10)	0.1587 (3)	0.79947 (8)	0.0166 (3)
H5A	0.7985	0.1370	0.8506	0.020*
H5B	0.8060	-0.0318	0.7847	0.020*
C6	0.69101 (10)	0.2559 (3)	0.73607 (8)	0.0153 (3)
C7	0.64731 (10)	0.1640 (3)	0.65713 (8)	0.0158 (3)
C8	0.55387 (10)	0.2626 (3)	0.60037 (9)	0.0184 (3)
H8A	0.5238	0.1988	0.5471	0.022*
C9	0.50331 (11)	0.4500 (3)	0.61885 (9)	0.0201 (3)
C10	0.54813 (11)	0.5407 (3)	0.69713 (9)	0.0211 (3)
H10A	0.5147	0.6698	0.7109	0.025*
C11	0.64099 (11)	0.4466 (3)	0.75590 (9)	0.0180 (3)
C12	0.69872 (11)	-0.0344 (3)	0.63240 (9)	0.0209 (3)
H12A	0.6596	-0.0557	0.5745	0.031*
H12B	0.7076	-0.2234	0.6572	0.031*
H12C	0.7623	0.0469	0.6494	0.031*
C13	0.40305 (12)	0.5556 (4)	0.55574 (10)	0.0287 (4)
H13A	0.3996	0.7646	0.5607	0.043*
H13B	0.3546	0.4608	0.5624	0.043*
H13C	0.3899	0.5098	0.5032	0.043*
C14	0.68632 (12)	0.5548 (4)	0.83981 (9)	0.0251 (3)
H14A	0.6451	0.7057	0.8406	0.038*
H14B	0.7509	0.6340	0.8592	0.038*
H14C	0.6923	0.3951	0.8741	0.038*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01899 (17)	0.02010 (18)	0.01301 (16)	-0.00305 (13)	0.00974 (14)	0.00054 (13)
O1	0.0200 (5)	0.0210 (5)	0.0134 (5)	-0.0015 (4)	0.0094 (4)	0.0009 (4)
N1	0.0188 (6)	0.0209 (6)	0.0125 (5)	-0.0052 (5)	0.0083 (5)	-0.0013 (4)
N2	0.0174 (6)	0.0170 (6)	0.0114 (5)	-0.0037 (4)	0.0082 (5)	-0.0004 (4)
C1	0.0201 (7)	0.0230 (8)	0.0167 (7)	-0.0076 (6)	0.0087 (6)	-0.0021 (6)
C2	0.0192 (7)	0.0230 (7)	0.0183 (7)	-0.0054 (6)	0.0099 (6)	0.0001 (6)
C3	0.0164 (6)	0.0145 (6)	0.0136 (6)	0.0002 (5)	0.0089 (5)	0.0007 (5)
C4	0.0160 (6)	0.0134 (6)	0.0139 (6)	0.0019 (5)	0.0077 (5)	0.0002 (5)
C5	0.0185 (7)	0.0151 (7)	0.0144 (6)	-0.0022 (5)	0.0079 (5)	0.0004 (5)
C6	0.0169 (6)	0.0133 (6)	0.0161 (6)	-0.0020 (5)	0.0092 (5)	0.0002 (5)
C7	0.0182 (6)	0.0125 (6)	0.0168 (6)	-0.0024 (5)	0.0097 (6)	-0.0009 (5)
C8	0.0181 (7)	0.0188 (7)	0.0155 (7)	-0.0037 (5)	0.0074 (6)	-0.0013 (5)

C9	0.0176 (7)	0.0211 (7)	0.0221 (7)	0.0000 (5)	0.0111 (6)	0.0034 (6)
C10	0.0217 (7)	0.0220 (8)	0.0240 (8)	0.0012 (6)	0.0155 (6)	0.0001 (6)
C11	0.0210 (7)	0.0176 (7)	0.0186 (7)	-0.0032 (5)	0.0130 (6)	-0.0020 (5)
C12	0.0245 (7)	0.0178 (7)	0.0169 (7)	0.0015 (6)	0.0091 (6)	-0.0020 (5)
C13	0.0211 (8)	0.0375 (10)	0.0267 (8)	0.0061 (7)	0.0125 (7)	0.0083 (7)
C14	0.0294 (8)	0.0288 (9)	0.0211 (8)	-0.0019 (7)	0.0164 (7)	-0.0051 (6)

Geometric parameters (Å, °)

S1—C2	1.7227 (16)	C7—C12	1.506 (2)
S1—C3	1.7267 (14)	C8—C9	1.386 (2)
O1—C4	1.2230 (17)	C8—H8A	0.9500
N1—C3	1.3119 (18)	C9—C10	1.389 (2)
N1—C1	1.3840 (19)	C9—C13	1.509 (2)
N2—C4	1.3709 (18)	C10—C11	1.394 (2)
N2—C3	1.3875 (18)	C10—H10A	0.9500
N2—H1	0.8794	C11—C14	1.510 (2)
C1—C2	1.351 (2)	C12—H12A	0.9800
C1—H1A	0.9500	C12—H12B	0.9800
C2—H2A	0.9500	C12—H12C	0.9800
C4—C5	1.516 (2)	C13—H13A	0.9800
C5—C6	1.519 (2)	C13—H13B	0.9800
C5—H5A	0.9900	C13—H13C	0.9800
C5—H5B	0.9900	C14—H14A	0.9800
C6—C7	1.404 (2)	C14—H14B	0.9800
C6—C11	1.405 (2)	C14—H14C	0.9800
C7—C8	1.397 (2)		
C2—S1—C3	88.58 (7)	C9—C8—H8A	118.7
C3—N1—C1	108.98 (12)	C7—C8—H8A	118.7
C4—N2—C3	122.51 (12)	C8—C9—C10	118.04 (14)
C4—N2—H1	120.3	C8—C9—C13	121.07 (14)
C3—N2—H1	117.2	C10—C9—C13	120.89 (15)
C2—C1—N1	116.28 (14)	C9—C10—C11	121.49 (14)
C2—C1—H1A	121.9	C9—C10—H10A	119.3
N1—C1—H1A	121.9	C11—C10—H10A	119.3
C1—C2—S1	110.20 (11)	C10—C11—C6	119.66 (13)
C1—C2—H2A	124.9	C10—C11—C14	119.06 (14)
S1—C2—H2A	124.9	C6—C11—C14	121.28 (14)
N1—C3—N2	120.93 (12)	C7—C12—H12A	109.5
N1—C3—S1	115.96 (11)	C7—C12—H12B	109.5
N2—C3—S1	123.11 (10)	H12A—C12—H12B	109.5
O1—C4—N2	121.68 (13)	C7—C12—H12C	109.5
O1—C4—C5	122.36 (13)	H12A—C12—H12C	109.5
N2—C4—C5	115.95 (12)	H12B—C12—H12C	109.5
C4—C5—C6	111.57 (12)	C9—C13—H13A	109.5
C4—C5—H5A	109.3	C9—C13—H13B	109.5
C6—C5—H5A	109.3	H13A—C13—H13B	109.5

C4—C5—H5B	109.3	C9—C13—H13C	109.5
C6—C5—H5B	109.3	H13A—C13—H13C	109.5
H5A—C5—H5B	108.0	H13B—C13—H13C	109.5
C7—C6—C11	119.71 (13)	C11—C14—H14A	109.5
C7—C6—C5	120.39 (13)	C11—C14—H14B	109.5
C11—C6—C5	119.87 (13)	H14A—C14—H14B	109.5
C8—C7—C6	118.59 (13)	C11—C14—H14C	109.5
C8—C7—C12	119.60 (13)	H14A—C14—H14C	109.5
C6—C7—C12	121.81 (13)	H14B—C14—H14C	109.5
C9—C8—C7	122.52 (14)		
C3—N1—C1—C2	-0.6 (2)	C5—C6—C7—C8	-178.83 (13)
N1—C1—C2—S1	0.76 (19)	C11—C6—C7—C12	178.50 (13)
C3—S1—C2—C1	-0.54 (13)	C5—C6—C7—C12	0.6 (2)
C1—N1—C3—N2	179.97 (13)	C6—C7—C8—C9	0.8 (2)
C1—N1—C3—S1	0.13 (17)	C12—C7—C8—C9	-178.67 (14)
C4—N2—C3—N1	174.78 (13)	C7—C8—C9—C10	-0.2 (2)
C4—N2—C3—S1	-5.40 (19)	C7—C8—C9—C13	179.08 (15)
C2—S1—C3—N1	0.23 (13)	C8—C9—C10—C11	-0.3 (2)
C2—S1—C3—N2	-179.60 (13)	C13—C9—C10—C11	-179.52 (15)
C3—N2—C4—O1	-0.3 (2)	C9—C10—C11—C6	0.1 (2)
C3—N2—C4—C5	-179.76 (13)	C9—C10—C11—C14	179.44 (14)
O1—C4—C5—C6	-49.20 (19)	C7—C6—C11—C10	0.5 (2)
N2—C4—C5—C6	130.23 (13)	C5—C6—C11—C10	178.43 (13)
C4—C5—C6—C7	92.65 (16)	C7—C6—C11—C14	-178.81 (14)
C4—C5—C6—C11	-85.24 (16)	C5—C6—C11—C14	-0.9 (2)
C11—C6—C7—C8	-0.9 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H1 \cdots N1 ⁱ	0.88	2.08	2.9623 (19)	175

Symmetry code: (i) $-x+2, -y+1, -z+2$.