Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(4-Chlorophenyl)-3-nitropyridin-2-amine

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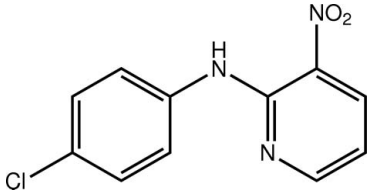
Received 24 October 2011; accepted 30 October 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.091; data-to-parameter ratio = 14.9.

In the title compound, $C_{11}H_8ClN_3O_2$, the presence of intramolecular N—H···O and C—H···N interactions help to establish an almost planar molecule [dihedral angle between the pyridine and benzene rings = 9.89 (8)° and r.m.s. deviation for all 17 non-H atoms = 0.120 Å]. Supramolecular tapes feature in the crystal packing whereby dimeric aggregates sustained by pairs of C—H···O interactions are connected by π–π interactions occurring between translationally related pyridine rings and between translationally related benzene rings along the b axis [centroid–centroid distance = length of b axis = 3.8032 (4) Å].

Related literature

For the structure of a related pyrimidine amine derivative, see: Aznan Akhmad *et al.* (2010).



Experimental

Crystal data

$C_{11}H_8ClN_3O_2$ M_r = 249.65
Monoclinic, $C2/c$
a = 30.472 (3) Å
b = 3.8032 (4) Å
c = 21.300 (2) Å
β = 123.153 (1)°
V = 2066.7 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 100 K
0.40 × 0.15 × 0.05 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.869, T_{max} = 0.982
8919 measured reflections
2347 independent reflections
1912 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.091
S = 1.00
2347 reflections
158 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*n*···O1	0.87 (2)	1.91 (2)	2.6280 (18)	138.2 (17)
C7—H7···N2	0.95	2.31	2.909 (2)	120
C3—H3···O2^{i}	0.95	2.48	3.340 (3)	152

Symmetry code: (i) $-x, -y+3, -z+1$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank the University of Malaya (grant No. RG027/09AFR) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6473).

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supporting information

Acta Cryst. (2011). E**67**, o3176 [https://doi.org/10.1107/S1600536811045570]

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S1. Comment

In connection with synthetic and structural studies of nitro-pyridine/pyrimidine derivatives (Aznan Akhmad *et al.*, 2010), the title compound, (I), was investigated. A small twist is evident in (I), Fig. 1, as seen in the value of the dihedral angle between the pyridyl and benzene rings of 9.89 (8) Å. The nitro group is co-planar with the pyridyl ring to which it is connected: the O1—N3—C2—C1 torsion angle is 5.0 (2)°. The observed conformation is stabilized by an intramolecular N—H···O hydrogen bond as well as a C—H···N interaction, Table 1. Overall, the molecule is close to planar with the r.m.s. deviation for all 17 non-hydrogen atoms being 0.120 Å.

In the crystal structure, centrosymmetrically related molecules are connected into dimeric aggregates *via* C—H···O interactions, Table 1. These are connected into a supramolecular tape along the *b* axis by *π*–*π* interactions between translationally related pyridyl rings and between translationally related benzene rings with the centroid···centroid separation corresponding to the length of the *b* axis, *i.e.* 3.8032 (4) Å, Fig. 2. The columns are connected by weak C—H···Cl [closest contact: C5—H5···Cl1^{i} = 3.97 Å, C5···Cl1^{i} = 3.6112 (19) Å and angle at H5 = 124° for *i*: 1/2 - *x*, 3/2 - *y*, 1 - *z*] and Cl···Cl [Cl1···Cl1^{ii} = 3.4366 (7) Å for *ii*: 1/2 - *x*, -1/2 + *y*, 1/2 - *z*] contacts. A view of the unit-cell contents is given in Fig. 3.

S2. Experimental

2-Chloro-3-nitro-pyridine (0.906 g, 0.0057 mol) and *p*-chloroaniline (0.730 g, 0.0057 mol) were refluxed in ethanol (5 ml) for 4 h at 385 K. After cooling the mixture, the residue was dissolved in a minimum volume of water (10 ml) and extracted with ether (3 *x* 10 ml). The ethereal layer was washed with water and dried over anhydrous sodium sulfate. Evaporation gave a red solid and recrystallization from its diethyl ether solution yielded red-brown prisms of (I) after a few days.

S3. Refinement

Carbon-bound hydrogen atoms were placed at calculated positions (C—H 0.95 Å) and were treated as riding on their parent carbon atoms, with *U*(H) set to 1.2 times U_{eq}(C). The amine-H atom was refined with N—H = 0.86±0.01 Å with refined U_{iso}.

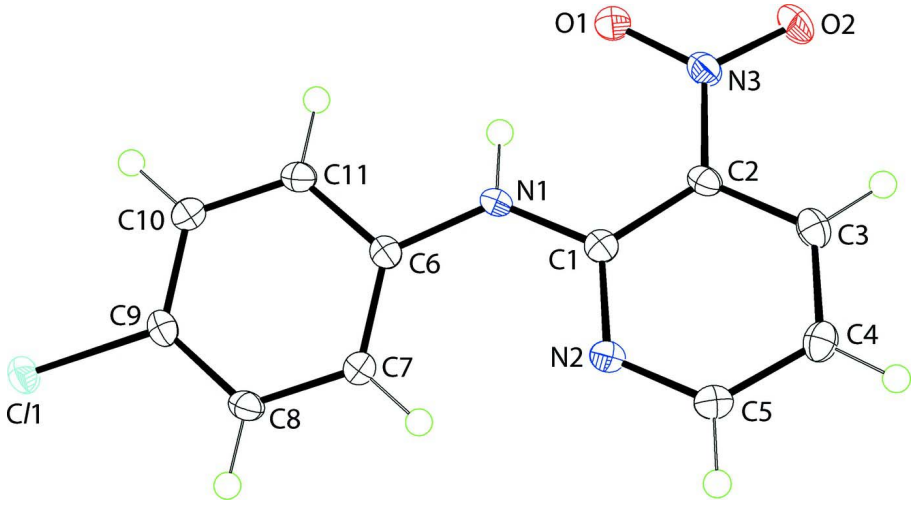

Figure 1

The molecular structure of (I) showing displacement ellipsoids at the 50% probability level.

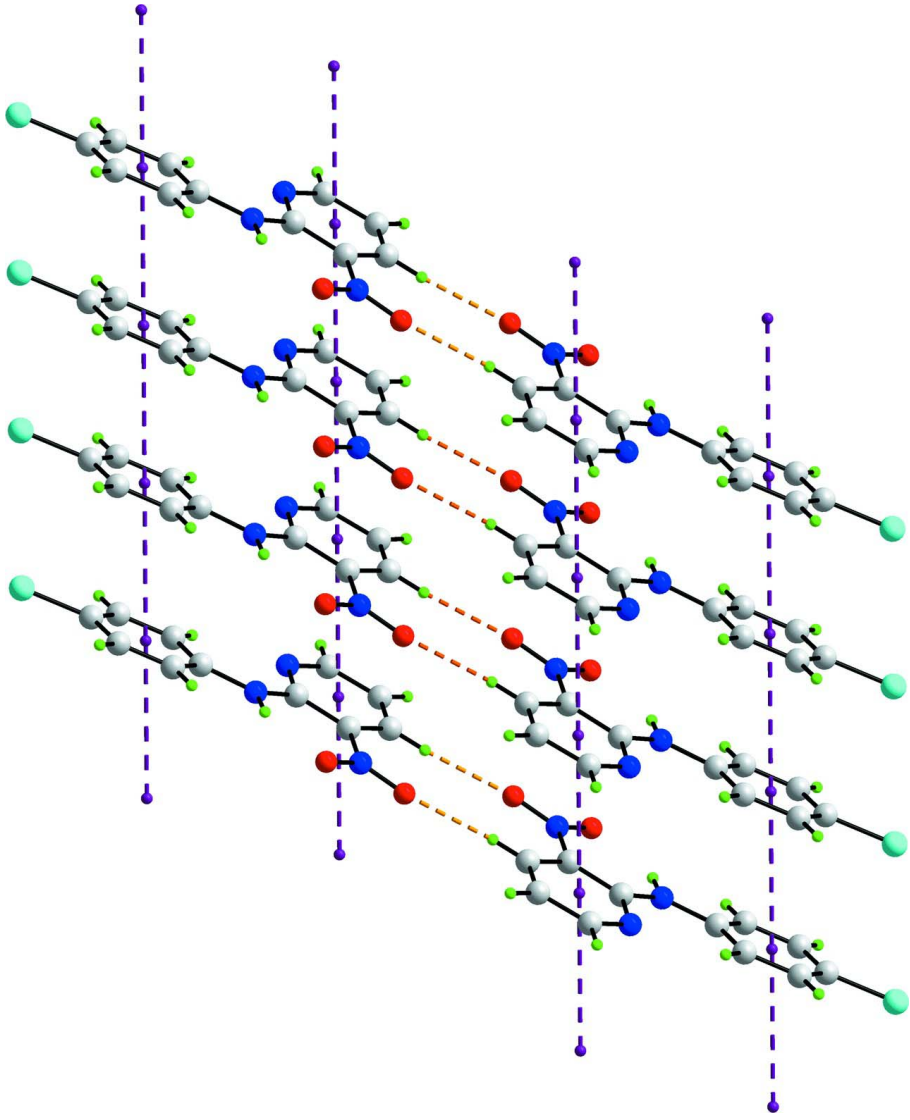

Figure 2

Supramolecular tape along the *b* axis in (I) sustained by C—H···O and π–π interactions shown as orange and purple dashed lines, respectively.

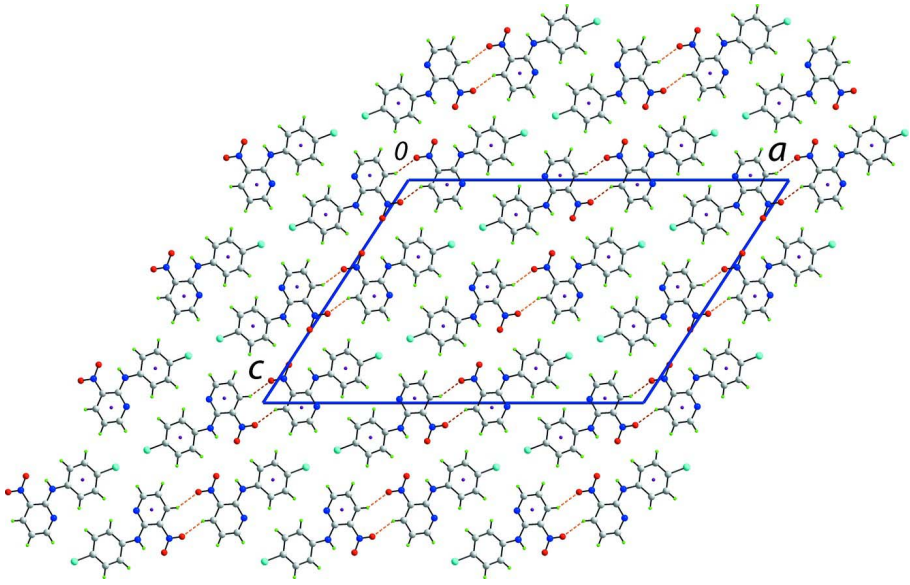

Figure 3

Unit-cell contents for (I) shown in projection down the *b* axis. The C—H···O interactions are shown as orange dashed lines.

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Crystal data

$C_{11}H_8ClN_3O_2$
$M_r = 249.65$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 30.472\ (3)$ Å
$b = 3.8032\ (4)$ Å
$c = 21.300\ (2)$ Å
$\beta = 123.153\ (1)°$
$V = 2066.7\ (4)$ Å^3
$Z = 8$
$F(000) = 1024$
$D_x = 1.605$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2586 reflections
$\theta = 2.3–28.0°$
$\mu = 0.36$ mm^{-1}
$T = 100$ K
Prism, red-brown
0.40 × 0.15 × 0.05 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.869$, $T_{max} = 0.982$
8919 measured reflections
2347 independent reflections
1912 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.6°$
$h = -36\rightarrow38$
$k = -4\rightarrow4$
$l = -27\rightarrow27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.091$
$S = 1.00$
2347 reflections
158 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 atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2 + 1.1995P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

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^ > 2σ(*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}
Cl1	0.223442 (15)	0.15167 (11)	0.28841 (2)	0.01760 (13)
O1	0.00287 (5)	1.1095 (3)	0.32850 (6)	0.0239 (3)
O2	−0.01657 (5)	1.3711 (4)	0.40019 (7)	0.0271 (3)
N1	0.09477 (5)	0.8069 (4)	0.38672 (8)	0.0151 (3)
H1n	0.0640 (8)	0.866 (5)	0.3481 (11)	0.021 (5)*
N2	0.14771 (5)	0.8205 (4)	0.51652 (7)	0.0155 (3)
N3	0.01356 (5)	1.1930 (4)	0.39171 (8)	0.0180 (3)
C1	0.10203 (6)	0.9037 (4)	0.45291 (9)	0.0141 (3)
C2	0.06265 (6)	1.0832 (4)	0.45711 (9)	0.0148 (3)
C3	0.07133 (7)	1.1672 (4)	0.52649 (9)	0.0175 (3)
H3	0.0452	1.2859	0.5296	0.021*
C4	0.11814 (7)	1.0768 (4)	0.59046 (9)	0.0181 (4)
H4	0.1252	1.1299	0.6388	0.022*
C5	0.15475 (6)	0.9047 (4)	0.58188 (9)	0.0172 (3)
H5	0.1872	0.8427	0.6261	0.021*
C6	0.12870 (6)	0.6443 (4)	0.36965 (9)	0.0137 (3)
C7	0.18163 (6)	0.5712 (4)	0.42051 (9)	0.0161 (3)
H7	0.1978	0.6249	0.4722	0.019*
C8	0.21055 (6)	0.4195 (4)	0.39510 (9)	0.0163 (3)
H8	0.2467	0.3708	0.4294	0.020*
C9	0.18699 (6)	0.3393 (4)	0.32026 (9)	0.0149 (3)
C10	0.13421 (6)	0.4065 (4)	0.26931 (9)	0.0161 (3)
H10	0.1181	0.3478	0.2179	0.019*
C11	0.10550 (6)	0.5593 (4)	0.29426 (9)	0.0158 (3)
H11	0.0694	0.6076	0.2596	0.019*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0176 (2)	0.0184 (2)	0.0213 (2)	0.00078 (16)	0.01348 (17)	−0.00104 (16)
O1	0.0167 (6)	0.0340 (8)	0.0164 (6)	0.0051 (5)	0.0062 (5)	−0.0024 (5)
O2	0.0182 (6)	0.0350 (8)	0.0279 (7)	0.0118 (6)	0.0125 (5)	−0.0006 (6)
N1	0.0103 (7)	0.0182 (7)	0.0148 (6)	0.0020 (6)	0.0056 (6)	−0.0004 (6)

N2	0.0130 (7)	0.0168 (7)	0.0157 (6)	0.0005 (6)	0.0071 (6)	0.0011 (5)
N3	0.0148 (7)	0.0186 (7)	0.0213 (7)	0.0018 (6)	0.0103 (6)	−0.0002 (6)
C1	0.0148 (8)	0.0107 (8)	0.0176 (7)	−0.0025 (6)	0.0094 (6)	−0.0004 (6)
C2	0.0115 (8)	0.0141 (8)	0.0184 (8)	−0.0007 (6)	0.0079 (6)	−0.0003 (6)
C3	0.0197 (8)	0.0143 (8)	0.0234 (8)	0.0004 (7)	0.0148 (7)	0.0006 (6)
C4	0.0225 (9)	0.0167 (8)	0.0178 (8)	−0.0018 (7)	0.0128 (7)	−0.0008 (6)
C5	0.0157 (8)	0.0178 (9)	0.0161 (7)	−0.0009 (7)	0.0073 (6)	0.0013 (6)
C6	0.0145 (8)	0.0104 (8)	0.0178 (7)	−0.0013 (6)	0.0098 (6)	0.0000 (6)
C7	0.0157 (8)	0.0170 (8)	0.0152 (7)	−0.0004 (7)	0.0082 (7)	−0.0016 (6)
C8	0.0115 (8)	0.0174 (8)	0.0183 (8)	0.0000 (6)	0.0070 (6)	−0.0001 (6)
C9	0.0168 (8)	0.0114 (8)	0.0206 (8)	0.0000 (6)	0.0129 (7)	0.0004 (6)
C10	0.0163 (8)	0.0158 (8)	0.0149 (7)	−0.0015 (6)	0.0078 (6)	−0.0006 (6)
C11	0.0129 (8)	0.0148 (8)	0.0169 (7)	−0.0004 (6)	0.0065 (6)	0.0003 (6)

Geometric parameters (Å, º)

Cl1—C9	1.7400 (16)	C4—C5	1.389 (2)
O1—N3	1.2408 (18)	C4—H4	0.9500
O2—N3	1.2312 (18)	C5—H5	0.9500
N1—C1	1.353 (2)	C6—C11	1.393 (2)
N1—C6	1.412 (2)	C6—C7	1.393 (2)
N1—H1n	0.87 (2)	C7—C8	1.387 (2)
N2—C5	1.327 (2)	C7—H7	0.9500
N2—C1	1.346 (2)	C8—C9	1.378 (2)
N3—C2	1.440 (2)	C8—H8	0.9500
C1—C2	1.426 (2)	C9—C10	1.386 (2)
C2—C3	1.389 (2)	C10—C11	1.376 (2)
C3—C4	1.372 (2)	C10—H10	0.9500
C3—H3	0.9500	C11—H11	0.9500
C1—N1—C6	131.43 (14)	N2—C5—H5	117.6
C1—N1—H1n	113.1 (12)	C4—C5—H5	117.6
C6—N1—H1n	115.4 (12)	C11—C6—C7	119.45 (15)
C5—N2—C1	118.99 (14)	C11—C6—N1	114.64 (14)
O2—N3—O1	121.71 (14)	C7—C6—N1	125.90 (14)
O2—N3—C2	118.75 (13)	C8—C7—C6	119.47 (14)
O1—N3—C2	119.54 (13)	C8—C7—H7	120.3
N2—C1—N1	118.36 (14)	C6—C7—H7	120.3
N2—C1—C2	119.48 (14)	C9—C8—C7	120.22 (15)
N1—C1—C2	122.15 (14)	C9—C8—H8	119.9
C3—C2—C1	120.01 (15)	C7—C8—H8	119.9
C3—C2—N3	117.09 (14)	C8—C9—C10	120.84 (15)
C1—C2—N3	122.88 (14)	C8—C9—Cl1	120.15 (13)
C4—C3—C2	119.25 (15)	C10—C9—Cl1	119.02 (12)
C4—C3—H3	120.4	C11—C10—C9	119.08 (15)
C2—C3—H3	120.4	C11—C10—H10	120.5
C3—C4—C5	117.45 (15)	C9—C10—H10	120.5
C3—C4—H4	121.3	C10—C11—C6	120.93 (15)

C5—C4—H4	121.3	C10—C11—H11	119.5
N2—C5—C4	124.83 (15)	C6—C11—H11	119.5
C5—N2—C1—N1	−178.05 (15)	C1—N2—C5—C4	−0.2 (3)
C5—N2—C1—C2	0.7 (2)	C3—C4—C5—N2	−0.2 (3)
C6—N1—C1—N2	−4.5 (3)	C1—N1—C6—C11	175.03 (16)
C6—N1—C1—C2	176.74 (16)	C1—N1—C6—C7	−6.0 (3)
N2—C1—C2—C3	−0.7 (2)	C11—C6—C7—C8	0.9 (2)
N1—C1—C2—C3	177.97 (16)	N1—C6—C7—C8	−178.01 (15)
N2—C1—C2—N3	177.63 (14)	C6—C7—C8—C9	−0.5 (3)
N1—C1—C2—N3	−3.7 (3)	C7—C8—C9—C10	−0.3 (3)
O2—N3—C2—C3	4.2 (2)	C7—C8—C9—Cl1	179.57 (13)
O1—N3—C2—C3	−176.53 (15)	C8—C9—C10—C11	0.8 (2)
O2—N3—C2—C1	−174.27 (15)	Cl1—C9—C10—C11	−179.10 (13)
O1—N3—C2—C1	5.0 (2)	C9—C10—C11—C6	−0.4 (3)
C1—C2—C3—C4	0.3 (3)	C7—C6—C11—C10	−0.4 (2)
N3—C2—C3—C4	−178.19 (15)	N1—C6—C11—C10	178.60 (15)
C2—C3—C4—C5	0.2 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*n*···O1	0.87 (2)	1.91 (2)	2.6280 (18)	138.2 (17)
C7—H7···N2	0.95	2.31	2.909 (2)	120
C3—H3···O2^{i}	0.95	2.48	3.340 (3)	152

Symmetry code: (i) −*x*, −*y*+3, −*z*+1.