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1-(4-Chlorophenyl)-1*H*-1,2,4-triazol-5(4*H*)-one

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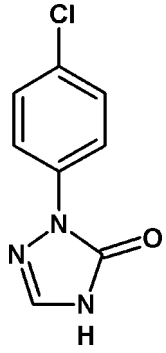
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.094; data-to-parameter ratio = 12.2.

In the title compound, $C_8H_6ClN_3O$, the dihedral angle between the 1,2,4-triazole and benzene rings is 4.60 (9)° and an intramolecular C—H···O interaction closes an S(6) ring. In the crystal, inversion dimers linked by pairs of N—H···O hydrogen bonds generate $R_2^2(8)$ loops and C—H···O interactions link the dimers into [100] chains. Weak π–π stacking interactions [centroid–centroid distance = 3.644 (1) Å] are also observed.

Related literature

For a related structure and background to 1,2,4-triazoles, see: Devarajegowda *et al.*(2012).



Experimental

Crystal data

$C_8H_6ClN_3O$
M_r = 195.61
Triclinic, $P\overline{1}$
a = 6.5791 (4) Å
b = 7.2663 (4) Å
c = 9.3342 (5) Å
α = 80.121 (4)°
β = 85.042 (4)°
γ = 70.235 (4)°
V = 413.52 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.42 mm^{-1}
T = 296 K
0.24 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: ψ scan (*SADABS*; Sheldrick, 2007)
T_{min} = 0.770, T_{max} = 1.000
5938 measured reflections
1438 independent reflections
1270 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.094
S = 1.06
1438 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5···O2^{i}	0.86	1.95	2.7924 (18)	166
C6—H6···O2^{ii}	0.93	2.53	3.360 (3)	149
C9—H9···O2	0.93	2.29	2.933 (2)	126

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $x+1, y, z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97*.

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7211).

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2014). E**70**, o499 [doi:10.1107/S1600536814006412]

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S1. Comment

As part of our onging studies of 1,2,4-triazoles (Devarajegowda *et al.*, 2012), we now describe the synthesis and structure of the title compound.

The asymmetric unit of 2-(4-chlorophenyl)-2,4-dihydro-3*H*-1,2,4-triazol- 3-one is shown in Fig. 1. The dihedral angle between the 1,2,4-triazol ring (N3/N4/N5/C6/C7) and the benzene ring (C8–C13) is 4.60 (9)°. In the crystal, inversion related N5—H5···O2 interactions generate an $R_2^2(8)$ ring pattern and link pairs of independent molecules into dimers and C6—H6···O interactions generate $R_2^2(10)$ ring motifs. $C_g(1)\pi$–$\pi C_g(2)$ interactions [centroid–centroid distance = 3.644 (1) Å] between 1,2,4 triazole (N3/N4/N5/C6/C7) and benzene (C8–C13) rings are also observed.

S2. Experimental

2-(4-Chlorophenyl)-2,4-dihydro-3*H*-1,2,4-triazol-3-one was refluxed with formamide at 453 K. After completion of the reaction, the reaction mixture was poured into ice cold water to recover the title compound, which was recrystallized from ethanol solution as colourless plates (m.p. 528 K).

S3. Refinement

All H atoms were positioned geometrically, with N—H = 0.86 Å, and C—H = 0.93 Å for aromatic H and refined using a riding model with U_{iso}(H) = 1.2U_{eq}(C, N) for aromatic and amide H.

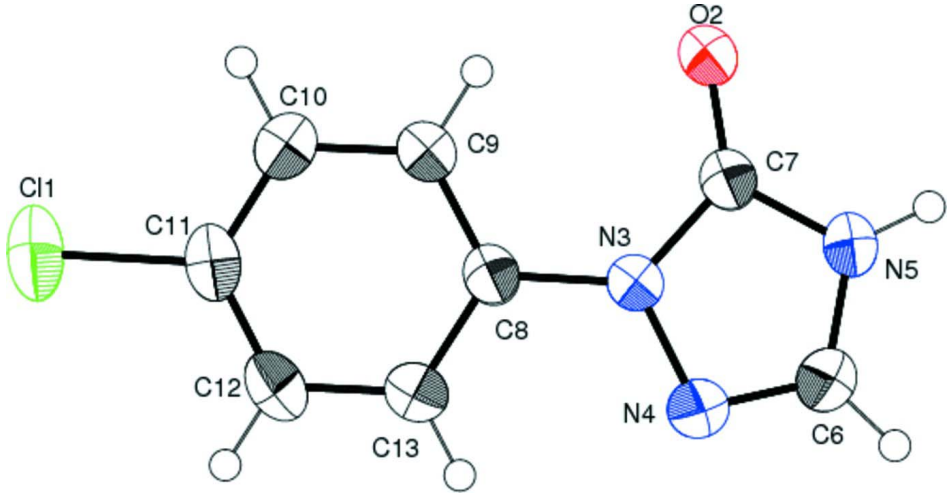

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

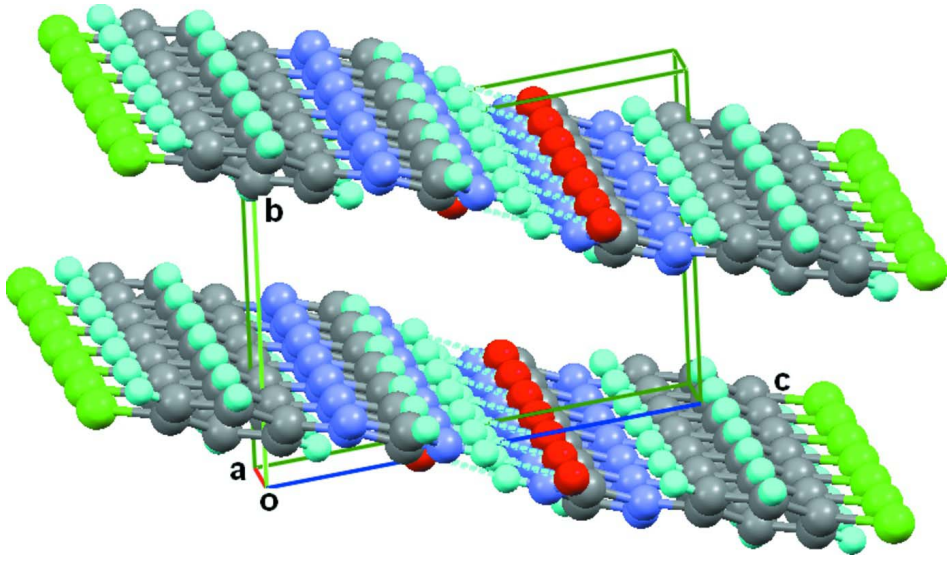

Figure 2

The packing of the title compound.

1-(4-Chlorophenyl)-1*H*-1,2,4-triazol-5(4*H*)-one

Crystal data

$C_8H_6ClN_3O$
M_r = 195.61
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.5791 (4) Å
b = 7.2663 (4) Å
c = 9.3342 (5) Å
α = 80.121 (4)°
β = 85.042 (4)°
γ = 70.235 (4)°
V = 413.52 (4) Å^3
Z = 2
$F(000)$ = 200
D_x = 1.571 Mg m^{-3}
Melting point: 528 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1438 reflections
θ = 2.2–25.0°
μ = 0.42 mm^{-1}
T = 296 K
Plate, colourless
0.24 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: ψ scan (*SADABS*; Sheldrick, 2007)
T_{min} = 0.770, T_{max} = 1.000
5938 measured reflections
1438 independent reflections
1270 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.0°, θ_{min} = 2.2°
h = −7→7
k = −8→8
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.094
S = 1.06
1438 reflections
118 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
$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.1038P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

Special details

Experimental. IR (KBr): 1686 (C=O), 3433 (NH); ^{1}H-NMR (400 MHz, DMSO-D$_6$, δ p.p.m.): 7.46–7.50 (d, 2H, ArH, J = 16 Hz), 7.90–7.94 (d, 2H, ArH, J = 16 Hz), 8.12 (s, 1H, C5H), 12.00 (s, 1H, NH); ^{13}C-NMR (100 MHz, DMSO-D$_6$, δ p.p.m.): 119.26, 128.77, 128.84, 136.66, 136.72, 152.17; MS (m/*z*, 70 eV): 197 (M^{2+}), 195 (M^+), 127, 125, 113, 111.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	−0.31296 (10)	0.62029 (8)	1.39673 (5)	0.0705 (2)
O2	−0.1420 (2)	0.9297 (2)	0.66886 (13)	0.0595 (4)
N3	0.1322 (2)	0.79679 (19)	0.83947 (14)	0.0385 (3)
N4	0.3528 (2)	0.7607 (2)	0.83651 (15)	0.0488 (4)
N5	0.2213 (2)	0.8940 (2)	0.62018 (15)	0.0457 (4)
H5	0.2196	0.9413	0.5291	0.055*
C11	−0.1807 (3)	0.6731 (2)	1.23269 (18)	0.0462 (4)
C10	−0.2983 (3)	0.7604 (3)	1.1128 (2)	0.0600 (5)
H10	−0.4479	0.7927	1.1186	0.072*
C9	−0.1960 (3)	0.8017 (3)	0.9812 (2)	0.0557 (5)
H9	−0.2767	0.8615	0.8986	0.067*
C8	0.0241 (2)	0.7544 (2)	0.97287 (16)	0.0369 (3)
C7	0.0477 (3)	0.8791 (2)	0.70550 (17)	0.0414 (4)
C12	0.0388 (3)	0.6240 (3)	1.2260 (2)	0.0600 (5)
H12	0.1183	0.5645	1.3092	0.072*
C13	0.1426 (3)	0.6632 (3)	1.0949 (2)	0.0563 (5)
H13	0.2924	0.6278	1.0894	0.068*
C6	0.3967 (3)	0.8216 (3)	0.70340 (19)	0.0500 (4)
H6	0.5350	0.8162	0.6684	0.060*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0853 (4)	0.0835 (4)	0.0407 (3)	−0.0336 (3)	0.0192 (2)	−0.0029 (2)
O2	0.0439 (7)	0.0937 (10)	0.0378 (6)	−0.0286 (7)	−0.0083 (5)	0.0143 (6)
N3	0.0342 (7)	0.0481 (7)	0.0320 (7)	−0.0137 (5)	−0.0030 (5)	−0.0012 (5)
N4	0.0351 (8)	0.0675 (9)	0.0393 (8)	−0.0140 (6)	−0.0019 (6)	−0.0020 (6)
N5	0.0429 (8)	0.0615 (8)	0.0319 (7)	−0.0210 (6)	0.0008 (6)	0.0018 (6)
C11	0.0586 (11)	0.0470 (9)	0.0333 (8)	−0.0202 (8)	0.0072 (7)	−0.0046 (7)
C10	0.0396 (9)	0.0893 (14)	0.0466 (10)	−0.0202 (9)	0.0037 (8)	−0.0030 (9)

C9	0.0403 (9)	0.0861 (13)	0.0344 (9)	−0.0181 (9)	−0.0046 (7)	0.0041 (8)
C8	0.0399 (8)	0.0389 (8)	0.0313 (8)	−0.0132 (6)	−0.0013 (6)	−0.0029 (6)
C7	0.0411 (9)	0.0499 (9)	0.0337 (8)	−0.0187 (7)	−0.0030 (7)	0.0011 (6)
C12	0.0579 (12)	0.0775 (13)	0.0346 (9)	−0.0167 (10)	−0.0070 (8)	0.0096 (8)
C13	0.0399 (9)	0.0786 (12)	0.0412 (10)	−0.0149 (8)	−0.0072 (8)	0.0087 (8)
C6	0.0377 (9)	0.0696 (11)	0.0405 (9)	−0.0180 (8)	0.0025 (7)	−0.0041 (8)

Geometric parameters (Å, º)

Cl1—C11	1.7444 (16)	C11—C12	1.364 (3)
O2—C7	1.237 (2)	C10—C9	1.385 (3)
N3—C7	1.367 (2)	C10—H10	0.9300
N3—N4	1.3833 (19)	C9—C8	1.369 (2)
N3—C8	1.419 (2)	C9—H9	0.9300
N4—C6	1.288 (2)	C8—C13	1.375 (2)
N5—C6	1.348 (2)	C12—C13	1.383 (3)
N5—C7	1.359 (2)	C12—H12	0.9300
N5—H5	0.8600	C13—H13	0.9300
C11—C10	1.352 (3)	C6—H6	0.9300
C7—N3—N4	111.34 (13)	C9—C8—C13	119.67 (15)
C7—N3—C8	128.99 (13)	C9—C8—N3	120.90 (14)
N4—N3—C8	119.63 (12)	C13—C8—N3	119.43 (15)
C6—N4—N3	103.86 (13)	O2—C7—N5	127.42 (15)
C6—N5—C7	107.98 (14)	O2—C7—N3	128.69 (15)
C6—N5—H5	126.0	N5—C7—N3	103.89 (13)
C7—N5—H5	126.0	C11—C12—C13	119.63 (17)
C10—C11—C12	120.83 (16)	C11—C12—H12	120.2
C10—C11—Cl1	119.12 (14)	C13—C12—H12	120.2
C12—C11—Cl1	120.04 (14)	C8—C13—C12	119.95 (17)
C11—C10—C9	119.95 (17)	C8—C13—H13	120.0
C11—C10—H10	120.0	C12—C13—H13	120.0
C9—C10—H10	120.0	N4—C6—N5	112.93 (15)
C8—C9—C10	119.94 (17)	N4—C6—H6	123.5
C8—C9—H9	120.0	N5—C6—H6	123.5
C10—C9—H9	120.0		
C7—N3—N4—C6	0.28 (18)	C6—N5—C7—N3	0.31 (18)
C8—N3—N4—C6	−177.50 (14)	N4—N3—C7—O2	179.93 (18)
C12—C11—C10—C9	−0.3 (3)	C8—N3—C7—O2	−2.6 (3)
Cl1—C11—C10—C9	−179.35 (16)	N4—N3—C7—N5	−0.37 (18)
C11—C10—C9—C8	−0.1 (3)	C8—N3—C7—N5	177.14 (14)
C10—C9—C8—C13	1.1 (3)	C10—C11—C12—C13	−0.3 (3)
C10—C9—C8—N3	−179.50 (17)	Cl1—C11—C12—C13	178.82 (16)
C7—N3—C8—C9	−2.1 (3)	C9—C8—C13—C12	−1.6 (3)
N4—N3—C8—C9	175.26 (15)	N3—C8—C13—C12	178.97 (17)
C7—N3—C8—C13	177.37 (16)	C11—C12—C13—C8	1.2 (3)
N4—N3—C8—C13	−5.3 (2)	N3—N4—C6—N5	−0.1 (2)

C6—N5—C7—O2	−179.98 (18)	C7—N5—C6—N4	−0.2 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5···O2^{i}	0.86	1.95	2.7924 (18)	166
C6—H6···O2^{ii}	0.93	2.53	3.360 (3)	149
C9—H9···O2	0.93	2.29	2.933 (2)	126

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