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1-(4-Chloro-2-fluoro-5-nitrophenyl)-4-difluoromethyl-3-methyl-1*H*-1,2,4-triazol-5(4*H*)-one

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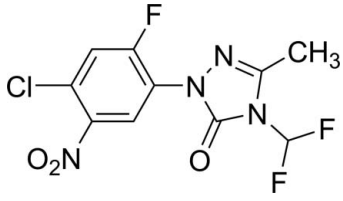
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.068; wR factor = 0.185; data-to-parameter ratio = 12.1.

In the title compound, $C_{10}H_6ClF_3N_4O_3$, the dihedral angle between the benzene ring and the triazolone ring is 59.9 (1)°, while the nitro substituent subtends an angle of 39.5 (1)° to the benzene ring plane. In the crystal, pairs of molecules form inversion dimers *via* C—H···O hydrogen bonds.

Related literature

For background to applications of this class of compound, see: Ager & Polsz (1996). For the synthesis and the use of the title compound in the production of herbicides, see: Goudar (1998). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{10}H_6ClF_3N_4O_3$
M_r = 322.64
Monoclinic, $P2_1/c$
a = 12.556 (3) Å
b = 14.800 (3) Å
c = 6.8760 (14) Å
β = 103.32 (3)°
V = 1243.4 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.899, T_{max} = 0.965
4877 measured reflections
2293 independent reflections
1589 reflections with $I > 2\sigma(I)$
R_{int} = 0.073
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.185
S = 1.01
2293 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10A···O1^{i}	0.98	2.32	3.190 (6)	148

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXS97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors thank the Center for Testing and Analysis, Nanjing University, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5217).

References

Ager, J. W. & Polsz, C. A. (1996). EP Patent No. 1273232.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Enraf–Nonius (1985). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
Goudar, J. S. (1998). US Patent No. 5756755.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2012). E**68**, o1374 [doi:10.1107/S1600536812013013]

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Comment

The title compound is an important intermediate used to synthesize the herbicide Carfentrazone-ethyl. It can also be used to synthesize other herbicides (Goudar, 1998), which are of wide interest for application to the control of broadleaf weeds and sedges (Ager & Polsz, 1996). We report here the crystal structure of the title compound, (I), which is of interest to us in this field.

The molecular structure of (I) is shown in Fig. 1. Bond distances in the molecule are normal (Allen *et al.*, 1987). The dihedral angle between the C1—C6 and N1/N3/C8/N2/C7 rings is 59.9 (1)° and the nitro substituent subtends an angle of 39.5 (1)° to the benzene ring plane.. In the crystal structure, molecules form inversion dimers via intermolecular C10—H10A···O1 hydrogen bonds (Table 1, Fig 2) .

Experimental

The title compound, (I) was prepared by a method reported in literature (Goudar, 1998). Crystals were obtained by dissolving (I) (0.2 g) in acetone (50 ml) and evaporating the solvent slowly at room temperature over 10 d.

Refinement

All H atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.93 Å for aromatic H and 0.96 Å for alkyl H, respectively. The U_{iso}(H) = xU_{eq}(C), where x = 1.2 for aromatic H, and x = 1.5 for alkyl H.

Computing details

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software* (Enraf–Nonius, 1985); data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXS97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

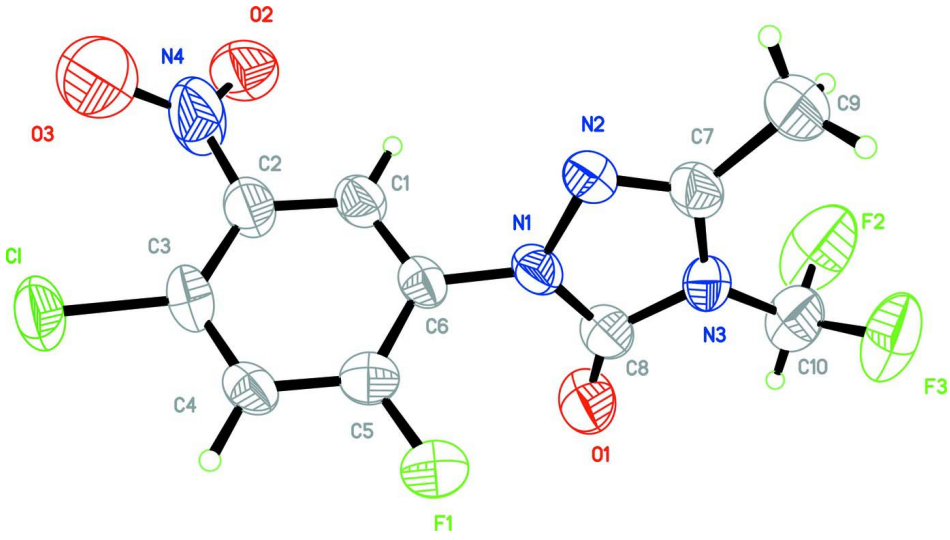

Figure 1

The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

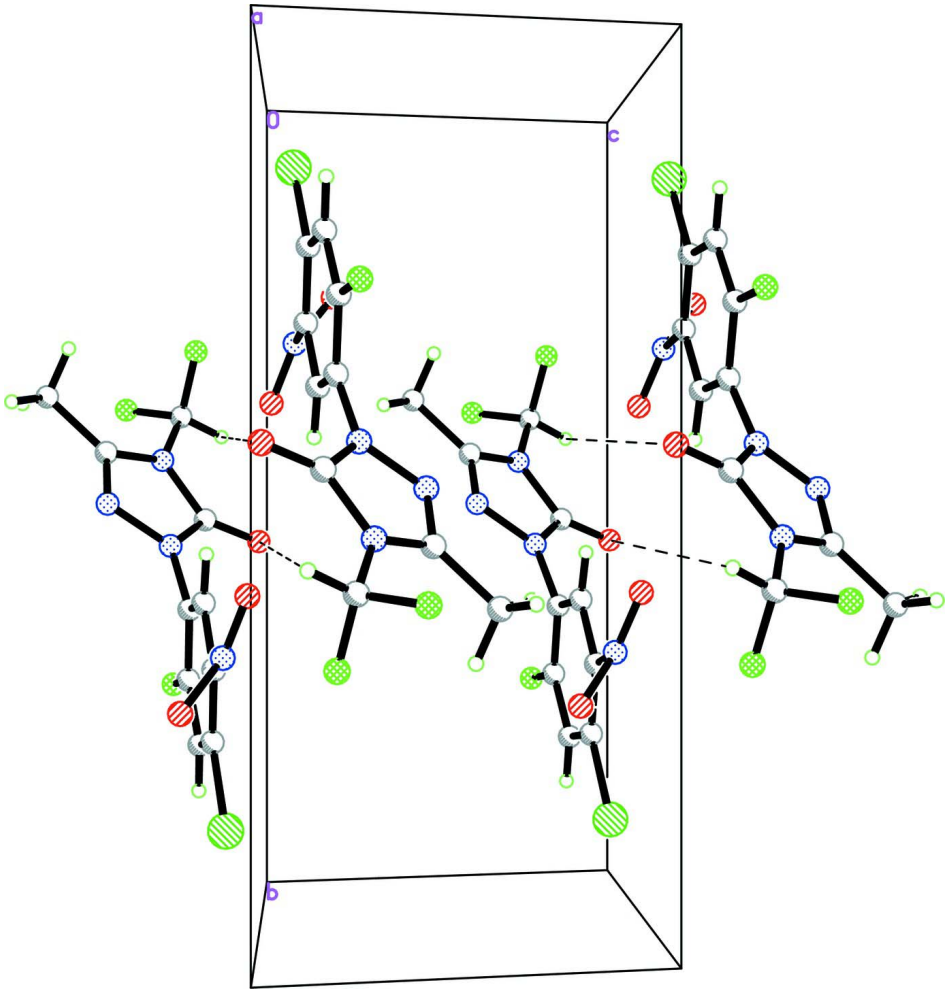

Figure 2

A packing diagram for (I) with hydrogen bonds drawn as dashed lines.

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$c = 6.8760$ (14) Å
$\beta = 103.32$ (3)°
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$Z = 4$

$F(000) = 648$
$D_x = 1.724$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.36$ mm^{-1}
$T = 293$ K
Block, colourless
0.30 × 0.20 × 0.10 mm

Data collection

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Radiation source: fine-focus sealed tube
Graphite monochromator

$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
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4877 measured reflections
2293 independent reflections
1589 reflections with $I > 2\sigma(I)$
$R_{int} = 0.073$
$\theta_{max} = 25.4°$, $\theta_{min} = 1.7°$
$h = -15 \rightarrow 14$
$k = -17 \rightarrow 17$
$l = 0 \rightarrow 8$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.185$
$S = 1.01$
2293 reflections
190 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
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.330P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.78$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

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}
Cl	0.47555 (10)	0.11625 (7)	0.08507 (17)	0.0670 (4)
F1	0.84029 (19)	0.26681 (16)	0.2492 (4)	0.0710 (7)
C1	0.5785 (3)	0.3747 (2)	0.1483 (5)	0.0470 (9)
H1A	0.5481	0.4322	0.1386	0.056*
N1	0.7581 (2)	0.43972 (19)	0.2491 (4)	0.0444 (7)
O1	0.8646 (3)	0.4432 (2)	0.0175 (5)	0.0709 (9)
O2	0.3580 (3)	0.3887 (3)	0.0249 (5)	0.0798 (10)
C2	0.5118 (3)	0.2996 (3)	0.1173 (7)	0.0580 (10)
F2	0.9214 (3)	0.6832 (2)	0.1945 (7)	0.1216 (14)
N2	0.7474 (2)	0.4912 (2)	0.4138 (5)	0.0481 (8)
N3	0.8789 (2)	0.5451 (2)	0.2821 (5)	0.0469 (8)
C3	0.5549 (3)	0.2129 (2)	0.1239 (6)	0.0521 (9)
F3	1.0420 (2)	0.6114 (2)	0.4004 (5)	0.0862 (9)
C4	0.6663 (3)	0.2027 (2)	0.1651 (5)	0.0414 (8)
H4A	0.6970	0.1454	0.1694	0.050*
N4	0.3939 (4)	0.3183 (3)	0.0870 (10)	0.113 (2)
C5	0.7323 (3)	0.2774 (2)	0.2001 (5)	0.0408 (8)
C6	0.6898 (3)	0.3641 (2)	0.1934 (5)	0.0379 (8)
C7	0.8207 (3)	0.5538 (2)	0.4266 (6)	0.0476 (9)
C8	0.8380 (3)	0.4709 (2)	0.1627 (5)	0.0471 (9)
C9	0.8423 (4)	0.6223 (3)	0.5868 (8)	0.0770 (14)

H9A	0.7916	0.6145	0.6708	0.116*
H9B	0.8337	0.6816	0.5287	0.116*
H9C	0.9157	0.6153	0.6653	0.116*
C10	0.9601 (3)	0.6014 (3)	0.2389 (7)	0.0626 (12)
H10A	0.9878	0.5764	0.1281	0.075*
O3	0.3405 (3)	0.2597 (4)	0.1869 (10)	0.143 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0777 (8)	0.0436 (6)	0.0810 (8)	−0.0207 (5)	0.0213 (6)	−0.0081 (5)
F1	0.0482 (13)	0.0551 (15)	0.109 (2)	0.0087 (11)	0.0167 (13)	−0.0066 (14)
C1	0.054 (2)	0.0352 (19)	0.053 (2)	0.0013 (16)	0.0148 (18)	0.0006 (15)
N1	0.0564 (18)	0.0368 (16)	0.0461 (16)	−0.0068 (14)	0.0245 (14)	−0.0097 (13)
O1	0.083 (2)	0.071 (2)	0.0702 (18)	−0.0221 (17)	0.0428 (17)	−0.0113 (16)
O2	0.062 (2)	0.077 (2)	0.097 (2)	0.0182 (17)	0.0115 (18)	0.0012 (19)
C2	0.045 (2)	0.042 (2)	0.081 (3)	−0.0005 (17)	0.003 (2)	−0.0040 (19)
F2	0.078 (2)	0.0610 (19)	0.216 (4)	−0.0003 (15)	0.015 (2)	0.064 (2)
N2	0.0489 (18)	0.0391 (17)	0.0596 (19)	−0.0017 (14)	0.0191 (15)	−0.0098 (14)
N3	0.0440 (17)	0.0391 (17)	0.0594 (18)	−0.0089 (13)	0.0157 (15)	−0.0011 (14)
C3	0.060 (2)	0.036 (2)	0.057 (2)	−0.0128 (18)	0.0075 (18)	−0.0031 (16)
F3	0.0552 (16)	0.085 (2)	0.107 (2)	−0.0209 (14)	−0.0060 (15)	0.0192 (16)
C4	0.064 (2)	0.0314 (17)	0.0366 (17)	0.0015 (16)	0.0283 (16)	−0.0008 (14)
N4	0.047 (2)	0.061 (3)	0.221 (6)	−0.007 (2)	0.011 (3)	−0.023 (4)
C5	0.046 (2)	0.047 (2)	0.0308 (16)	0.0043 (16)	0.0127 (14)	0.0011 (14)
C6	0.056 (2)	0.0337 (18)	0.0290 (16)	−0.0064 (15)	0.0188 (15)	−0.0035 (12)
C7	0.046 (2)	0.0327 (18)	0.060 (2)	0.0052 (16)	0.0041 (18)	−0.0031 (16)
C8	0.059 (2)	0.042 (2)	0.0453 (19)	−0.0057 (17)	0.0238 (18)	0.0005 (16)
C9	0.066 (3)	0.056 (3)	0.110 (4)	−0.002 (2)	0.021 (3)	−0.031 (3)
C10	0.059 (3)	0.040 (2)	0.089 (3)	−0.0079 (19)	0.017 (2)	0.023 (2)
O3	0.066 (2)	0.120 (4)	0.248 (7)	−0.023 (3)	0.044 (3)	−0.065 (4)

Geometric parameters (Å, º)

Cl—C3	1.729 (4)	N3—C7	1.368 (5)
F1—C5	1.329 (4)	N3—C8	1.397 (5)
C1—C6	1.368 (5)	N3—C10	1.401 (5)
C1—C2	1.379 (5)	C3—C4	1.370 (5)
C1—H1A	0.9300	F3—C10	1.336 (5)
N1—C8	1.359 (5)	C4—C5	1.369 (5)
N1—N2	1.397 (4)	C4—H4A	0.9300
N1—C6	1.408 (4)	N4—O3	1.373 (8)
O1—C8	1.196 (4)	C5—C6	1.387 (5)
O2—N4	1.176 (6)	C7—C9	1.475 (6)
C2—C3	1.389 (5)	C9—H9A	0.9600
C2—N4	1.473 (6)	C9—H9B	0.9600
F2—C10	1.315 (5)	C9—H9C	0.9600
N2—C7	1.294 (5)	C10—H10A	0.9800
C6—C1—C2	119.7 (3)	F1—C5—C6	118.7 (3)

C6—C1—H1A	120.1	C4—C5—C6	121.9 (3)
C2—C1—H1A	120.1	C1—C6—C5	118.6 (3)
C8—N1—N2	112.8 (3)	C1—C6—N1	119.8 (3)
C8—N1—C6	128.0 (3)	C5—C6—N1	121.3 (3)
N2—N1—C6	119.2 (3)	N2—C7—N3	111.9 (3)
C1—C2—C3	121.4 (4)	N2—C7—C9	123.3 (4)
C1—C2—N4	115.2 (4)	N3—C7—C9	124.7 (4)
C3—C2—N4	123.3 (4)	O1—C8—N1	128.7 (4)
C7—N2—N1	104.3 (3)	O1—C8—N3	128.7 (3)
C7—N3—C8	108.4 (3)	N1—C8—N3	102.6 (3)
C7—N3—C10	129.4 (3)	C7—C9—H9A	109.5
C8—N3—C10	122.0 (3)	C7—C9—H9B	109.5
C4—C3—C2	118.7 (3)	H9A—C9—H9B	109.5
C4—C3—Cl	117.7 (3)	C7—C9—H9C	109.5
C2—C3—Cl	123.5 (3)	H9A—C9—H9C	109.5
C5—C4—C3	119.7 (3)	H9B—C9—H9C	109.5
C5—C4—H4A	120.2	F2—C10—F3	105.3 (4)
C3—C4—H4A	120.2	F2—C10—N3	110.4 (4)
O2—N4—O3	123.4 (5)	F3—C10—N3	110.4 (3)
O2—N4—C2	120.4 (5)	F2—C10—H10A	110.2
O3—N4—C2	113.7 (5)	F3—C10—H10A	110.2
F1—C5—C4	119.4 (3)	N3—C10—H10A	110.2

C6—C1—C2—C3	−2.3 (6)	C8—N1—C6—C1	−123.1 (4)
C6—C1—C2—N4	174.9 (4)	N2—N1—C6—C1	57.8 (4)
C8—N1—N2—C7	0.3 (4)	C8—N1—C6—C5	62.6 (5)
C6—N1—N2—C7	179.5 (3)	N2—N1—C6—C5	−116.5 (3)
C1—C2—C3—C4	1.0 (6)	N1—N2—C7—N3	−0.9 (4)
N4—C2—C3—C4	−176.0 (5)	N1—N2—C7—C9	−177.2 (4)
C1—C2—C3—Cl	−179.7 (3)	C8—N3—C7—N2	1.2 (4)
N4—C2—C3—Cl	3.4 (7)	C10—N3—C7—N2	176.2 (4)
C2—C3—C4—C5	0.5 (6)	C8—N3—C7—C9	177.5 (4)
Cl—C3—C4—C5	−178.9 (2)	C10—N3—C7—C9	−7.5 (6)
C1—C2—N4—O2	25.3 (8)	N2—N1—C8—O1	−177.8 (4)
C3—C2—N4—O2	−157.6 (5)	C6—N1—C8—O1	3.1 (7)
C1—C2—N4—O3	−137.4 (5)	N2—N1—C8—N3	0.4 (4)
C3—C2—N4—O3	39.7 (8)	C6—N1—C8—N3	−178.7 (3)
C3—C4—C5—F1	176.9 (3)	C7—N3—C8—O1	177.3 (4)
C3—C4—C5—C6	−0.6 (5)	C10—N3—C8—O1	1.9 (6)
C2—C1—C6—C5	2.1 (5)	C7—N3—C8—N1	−0.9 (4)
C2—C1—C6—N1	−172.3 (3)	C10—N3—C8—N1	−176.3 (3)
F1—C5—C6—C1	−178.2 (3)	C7—N3—C10—F2	−59.3 (6)
C4—C5—C6—C1	−0.7 (5)	C8—N3—C10—F2	115.1 (5)
F1—C5—C6—N1	−3.9 (4)	C7—N3—C10—F3	56.7 (5)
C4—C5—C6—N1	173.6 (3)	C8—N3—C10—F3	−128.9 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*A*···O1^{i}	0.98	2.32	3.190 (6)	148

Symmetry code: (i) −*x*+2, −*y*+1, −*z*.