Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Difluoromethyl-3-methyl-1-phenyl-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) = 0.003 Å
R factor = 0.043
wR factor = 0.121
Data-to-parameter ratio = 13.8

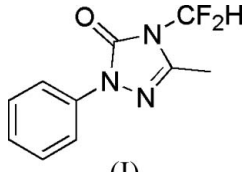
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 13 March 2006
Accepted 22 May 2006

In the crystal structure of the title compound, $C_{10}H_9F_2N_3O$, a derivative of triazolinone, there are intramolecular C—H···O and C—H···N and intermolecular C—H···F and C—H···O interactions.

Comment

1-Aryl-1,2,4-triazolin-5-one derivatives have herbicidal activity (Kajioka *et al.*, 1982) and can be used on soya beans (Keifer *et al.*, 1990) to prevent or destroy undesired plant growth (Theodoridis *et al.*, 1991). The title compound, (I), is an important intermediate of triazolinone and we report here the crystal structure.



(I)

The molecular structure of (I) is shown in Fig. 1, in which the dashed lines indicate intramolecular C—H···O and C—H···N hydrogen bonds. Selected bond lengths and angles are given in Table 1. Intermolecular C—H···F and C—H···O hydrogen bonds are shown in Fig. 2 (Table 2).

Experimental

A stirred solution of 4-difluoromethyl-3-methyl-1-phenyl-1,2,4-triazolin-5-one (0.12 mol), powdered potassium hydroxide (0.24 mol) and tetrabutylammonium bromide (0.012 mol) in tetrahydrofuran (500 ml) was cooled in an ice bath and chlorodifluoromethane was bubbled into the reaction mixture. The ice bath was removed and the bubbling was continued until condensation was observed on a dry-ice condenser attached to the reaction vessel. Upon completion of the addition, the reaction mixture was stirred at ambient temperature for 16 h. Additional powdered potassium hydroxide (0.012 mol) was added to the reaction mixture and it was again saturated with chlorodifluoromethane. The reaction mixture was stirred for 2 h then diluted with water. The mixture was extracted with diethyl ether and the combined extracts washed with water. The organic layer was dried with sodium sulfate and filtered. The filtrate was concentrated under reduced pressure to a residue. The residue was dissolved in methylene chloride and passed through a pad of silica gel. The eluate was concentrated under reduced pressure to a residual solid. The solid was recrystallized from dichloromethane–heptane (1:1). Crystals suitable for X-ray diffraction were obstained by slow evaporation of a $CHCl_3$ solution.

Crystal data

$C_{10}H_9F_2N_3O$
M_r = 225.20
Triclinic, $P\overline{1}$
a = 7.2910 (15) Å
b = 8.2100 (16) Å
c = 8.8710 (18) Å
α = 85.88 (3)°
β = 80.43 (3)°
γ = 81.71 (3)°
V = 517.53 (19) Å^3
Z = 2
D_x = 1.445 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 (2) K
Block, colourless
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.964, T_{max} = 0.988
2188 measured reflections
2019 independent reflections
1422 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
θ_{max} = 26.0°
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.121
S = 1.01
2019 reflections
146 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.06P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.131 (13)

Table 1
Selected geometric parameters (Å, °).

F1—C10	1.346 (2)	N1—C10	1.412 (2)
F2—C10	1.345 (2)	N2—C9	1.364 (2)
O—C9	1.219 (2)	N2—N3	1.396 (2)
N1—C8	1.381 (2)	N2—C3	1.423 (2)
N1—C9	1.392 (2)	N3—C8	1.286 (2)
C8—N1—C9	108.30 (15)	N3—C8—N1	111.07 (16)
C8—N1—C10	130.39 (16)	N3—C8—C7	124.93 (18)
C9—N1—C10	121.29 (15)	N1—C8—C7	124.00 (18)
C9—N2—N3	111.71 (14)	O—C9—N2	130.43 (17)
C9—N2—C3	128.48 (15)	O—C9—N1	126.32 (17)
N3—N2—C3	119.80 (14)	N2—C9—N1	103.24 (15)
C8—N3—N2	105.67 (14)	F2—C10—F1	105.52 (16)
C4—C3—N2	120.75 (17)	F2—C10—N1	110.18 (17)
C2—C3—N2	119.43 (17)	F1—C10—N1	110.13 (16)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···N3	0.93	2.46	2.795 (3)	102
C4—H4A···O	0.93	2.32	2.952 (2)	125
C7—H7C···F1^{i}	0.96	2.49	3.368 (3)	153
C10—H10A···O	0.98	2.46	2.857 (2)	104
C10—H10A···O^{ii}	0.98	2.30	3.100 (2)	138

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

All H atoms were positioned geometrically at distances of 0.93–0.97 Å and included in the refinement in a riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo,1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXL97*.

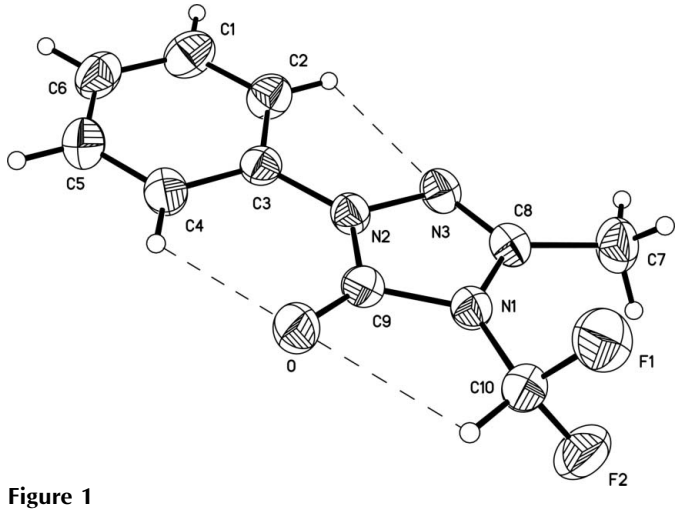


Figure 1
A view of the molecular structure of (I), showing displacement ellipsoids drawn at the 30% probability level. Dashed lines indicate C—H···O and C—H···N hydrogen bonds.

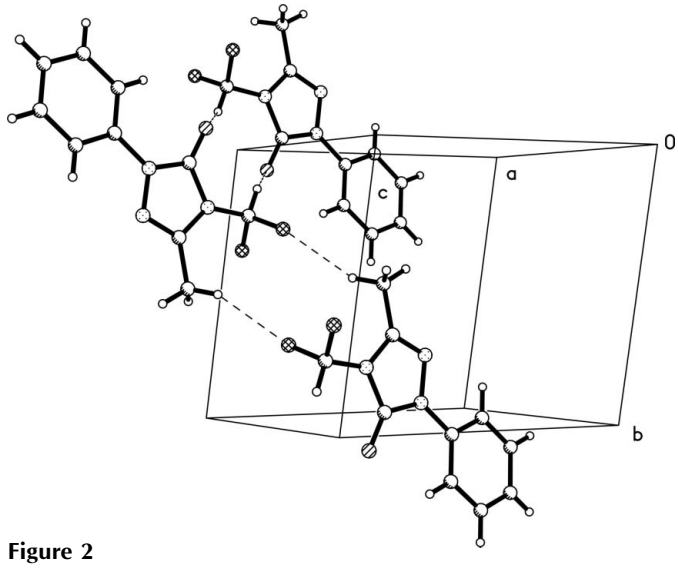


Figure 2
The crystal structure of (I). Dashed lines indicate C—H···O and C—H···F hydrogen bonds.

Author thank the Center of Test and Analysis, Nanjing University for the support.

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