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2-[3-(Trifluoromethyl)phenyl]perhydro-1,2,4-triazolo[1,2-*a*]pyridazine-1,3-dione

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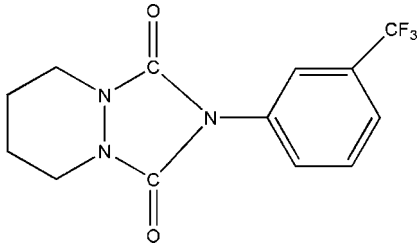
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.056; wR factor = 0.148; data-to-parameter ratio = 11.0.

In the title compound, $C_{13}H_{12}F_3N_3O_2$, the tetrahydropyridazine ring adopts a chair conformation. The amide ring is twisted away from the attached benzene ring by 27.3 (2)°. The F atoms are disordered over two positions, with site occupancy factors of 0.52 (2) and 0.48 (2).

Related literature

For related literature, see: Li *et al.* (2007).



Experimental

Crystal data

$C_{13}H_{12}F_3N_3O_2$
M_r = 299.26
Triclinic, $P\overline{1}$
a = 6.8348 (9) Å
b = 8.0756 (11) Å
c = 12.0770 (17) Å
α = 90.994 (2)°
β = 98.153 (2)°
γ = 95.630 (3)°
V = 656.34 (15) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 (2) K
0.50 × 0.37 × 0.30 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.751, T_{max} = 1.000
(expected range = 0.722–0.961)
3486 measured reflections
2409 independent reflections
1887 reflections with $I > 2\sigma(I)$
R_{int} = 0.131

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.148
S = 0.97
2409 reflections
219 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CS2041).

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supplementary materials

Acta Cryst. (2007). E**63**, o3840 [**doi:10.1107/S1600536807039748**]

2-[3-(Trifluoromethyl)phenyl]perhydro-1,2,4-triazolo[1,2-*a*]pyridazine-1,3-dione

Z.-G. Sun, X.-Q. Li, W. Xu and X.-H. Du

Comment

The title compound, (I), shows pesticidal activity. X-ray analysis was undertaken in order to establish its structure. The tetrahydropyridazine ring adopts a chair conformation and the five-membered ring lies close to the plane of the molecule. The C1—N1—C7—C12 torsion angle is 27.3 (2)°.

Experimental

Perhydropyridazine-1-carboxylic acid (3-trifluoromethylphenyl)-amide was prepared as described by Li *et al.* (2007). To a solution of perhydropyridazine-1-carboxylic acid (3-trifluoromethylphenyl)-amide (1.37 g, 5 mmol) in 1.2-dichloroethane (10 ml) were added the solution of bis(trichloromethyl)carbonate (0.59 g, 2 mmol) and pyridine (0.79 g, 10 mmol) in 1.2-dichloroethane (10 ml). The mixture was stirred at room temperature for 24 h. After the completion of the reaction, the mixture was washed with water and extracted with diethyl ether. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (eluant: petroleum ether-ethyl acetate, 1:1). Single crystals were obtained by slow evaporation of a petroleum ether-ethyl acetate solution (*v*/*v*: 1/1) (m.p. 417–418 K).

Refinement

All H atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and refined using a riding model with U_{iso}(H) = 1.2U_{eq}(C). The trifluoromethyl group was treated as disordered between two orientations with refined occupancies of 0.52 (2) and 0.48 (2), respectively. All C—F bonds lengths were restrained to 1.33 (1) Å and the displacement parameters of the disordered F atoms were restrained to an approximately isotropic behaviour. The large values of atomic displacement parameters for the disordered F atoms indicate further unresolved disorder of the trifluoromethyl group. The high value of R_{int} is due to the poor quality of the crystal.

Figures

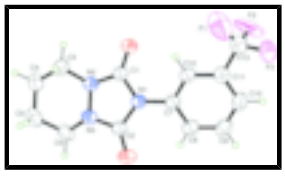

Fig. 1. The molecular structure of (I), showing the atom-labelling scheme with only one disorder component of the –CF3 group. Displacement ellipsoids are drawn at the 30% probability level.

2-[3-(Trifluoromethyl)phenyl]perhydro-1,2,4-triazolo[1,2-*a*]pyridazine-1,3-dione

Crystal data

$C_{13}H_{12}F_3N_3O_2$ F_{000} = 308

M_r = 299.26	D_x = 1.514 Mg m^{-3}
Triclinic, $P\bar{1}$	Melting point: 417 K
a = 6.8348 (9) Å	Mo $K\alpha$ radiation λ = 0.71073 Å
b = 8.0756 (11) Å	Cell parameters from 1670 reflections
c = 12.0770 (17) Å	θ = 6.0–53.0º
α = 90.994 (2)º	μ = 0.13 mm^{-1}
β = 98.153 (2)º	T = 293 (2) K
γ = 95.630 (3)º	Prism, colourless
V = 656.34 (15) Å^3	0.50 × 0.37 × 0.30 mm
Z = 2	

Data collection

Bruker SMART APEX diffractometer	2409 independent reflections
Radiation source: fine-focus sealed tube	1887 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.131
T = 293(2) K	θ_{max} = 25.5º
φ and ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −7→8
T_{min} = 0.751, T_{max} = 1.000	k = −9→9
3486 measured reflections	l = −14→14

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.056	H-atom parameters constrained
$wR(F^2)$ = 0.148	$w = 1/[\sigma^2(F_o^2) + (0.0885P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.97	$(\Delta/\sigma)_{max}$ = 0.021
2409 reflections	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
219 parameters	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
6 restraints	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.37 (3)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
F1	0.0262 (13)	0.720 (3)	0.0498 (10)	0.156 (5)	0.48 (2)
F2	−0.188 (2)	0.8924 (8)	0.0078 (9)	0.145 (4)	0.48 (2)
F3	−0.2579 (15)	0.6501 (15)	−0.0441 (6)	0.095 (3)	0.48 (2)

F1'	0.0267 (11)	0.7751 (13)	0.0497 (7)	0.099 (3)	0.52 (2)
F2'	−0.2280 (14)	0.8564 (18)	−0.0211 (9)	0.151 (4)	0.52 (2)
F3'	−0.185 (3)	0.599 (2)	−0.0220 (14)	0.164 (5)	0.52 (2)
O1	0.1920 (2)	0.91691 (18)	0.39839 (12)	0.0596 (5)	
O2	−0.2422 (2)	0.56464 (18)	0.56622 (13)	0.0639 (5)	
N1	−0.0668 (2)	0.73406 (17)	0.45001 (12)	0.0402 (4)	
N2	0.1823 (2)	0.83107 (19)	0.57773 (13)	0.0468 (4)	
N3	0.0621 (2)	0.70819 (19)	0.62498 (13)	0.0478 (4)	
C1	0.1134 (3)	0.8360 (2)	0.46708 (15)	0.0429 (5)	
C2	−0.1003 (3)	0.6587 (2)	0.54966 (16)	0.0444 (5)	
C3	0.3904 (3)	0.8445 (3)	0.62633 (17)	0.0532 (5)	
H3A	0.4637	0.9350	0.5931	0.064*	
H3B	0.4471	0.7422	0.6119	0.064*	
C4	0.4049 (3)	0.8771 (3)	0.75127 (17)	0.0573 (6)	
H4A	0.5414	0.8740	0.7860	0.069*	
H4B	0.3666	0.9872	0.7651	0.069*	
C5	0.2722 (4)	0.7491 (3)	0.80320 (17)	0.0581 (6)	
H5A	0.2747	0.7795	0.8815	0.070*	
H5B	0.3234	0.6416	0.7995	0.070*	
C6	0.0601 (3)	0.7350 (3)	0.74499 (16)	0.0558 (5)	
H6A	−0.0170	0.6425	0.7738	0.067*	
H6B	0.0001	0.8362	0.7582	0.067*	
C7	−0.1960 (3)	0.7115 (2)	0.34650 (15)	0.0397 (4)	
C8	−0.3976 (3)	0.6728 (2)	0.34434 (18)	0.0488 (5)	
H8	−0.4502	0.6599	0.4110	0.059*	
C9	−0.5213 (3)	0.6532 (2)	0.24317 (19)	0.0550 (5)	
H9	−0.6568	0.6252	0.2421	0.066*	
C10	−0.4459 (3)	0.6747 (2)	0.14363 (18)	0.0533 (5)	
H10	−0.5299	0.6633	0.0757	0.064*	
C11	−0.2447 (3)	0.7132 (2)	0.14634 (16)	0.0465 (5)	
C12	−0.1173 (3)	0.7311 (2)	0.24693 (15)	0.0431 (5)	
H12	0.0187	0.7558	0.2478	0.052*	
C13	−0.1607 (4)	0.7390 (3)	0.03994 (19)	0.0678 (7)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.080 (6)	0.239 (15)	0.068 (4)	0.077 (8)	0.036 (4)	0.025 (7)
F2	0.181 (12)	0.073 (4)	0.084 (5)	−0.024 (5)	0.068 (6)	0.030 (3)
F3	0.101 (4)	0.137 (6)	0.034 (2)	−0.039 (3)	0.004 (2)	−0.019 (3)
F1'	0.076 (5)	0.169 (5)	0.045 (3)	−0.041 (5)	0.018 (3)	0.015 (2)
F2'	0.131 (5)	0.163 (13)	0.069 (4)	0.063 (6)	0.011 (3)	0.082 (5)
F3'	0.162 (13)	0.134 (7)	0.110 (7)	−0.058 (7)	0.102 (7)	−0.063 (6)
O1	0.0457 (8)	0.0851 (10)	0.0442 (8)	−0.0150 (7)	0.0069 (6)	0.0176 (7)
O2	0.0486 (9)	0.0841 (10)	0.0579 (9)	−0.0113 (7)	0.0145 (7)	0.0199 (8)
N1	0.0305 (8)	0.0538 (9)	0.0377 (8)	0.0042 (6)	0.0091 (6)	0.0052 (6)
N2	0.0362 (9)	0.0661 (10)	0.0383 (8)	−0.0009 (7)	0.0095 (6)	0.0085 (7)
N3	0.0411 (9)	0.0651 (10)	0.0384 (9)	0.0008 (7)	0.0121 (7)	0.0101 (7)

C1	0.0336 (10)	0.0569 (11)	0.0391 (10)	0.0034 (7)	0.0083 (7)	0.0050 (8)
C2	0.0355 (10)	0.0562 (10)	0.0434 (10)	0.0045 (8)	0.0122 (8)	0.0092 (8)
C3	0.0367 (11)	0.0726 (13)	0.0494 (12)	0.0034 (9)	0.0046 (8)	0.0050 (9)
C4	0.0580 (13)	0.0657 (12)	0.0454 (11)	0.0077 (10)	−0.0032 (9)	0.0039 (9)
C5	0.0684 (15)	0.0678 (12)	0.0393 (11)	0.0164 (10)	0.0048 (9)	0.0073 (9)
C6	0.0599 (13)	0.0737 (13)	0.0381 (11)	0.0107 (10)	0.0174 (9)	0.0105 (9)
C7	0.0339 (9)	0.0452 (9)	0.0413 (10)	0.0065 (7)	0.0079 (7)	0.0036 (7)
C8	0.0353 (10)	0.0599 (11)	0.0533 (12)	0.0066 (8)	0.0122 (8)	0.0029 (9)
C9	0.0312 (10)	0.0677 (12)	0.0643 (13)	0.0034 (8)	0.0027 (9)	0.0014 (10)
C10	0.0460 (11)	0.0588 (12)	0.0514 (12)	0.0056 (8)	−0.0056 (9)	0.0014 (9)
C11	0.0458 (11)	0.0500 (10)	0.0431 (11)	0.0041 (8)	0.0043 (8)	0.0031 (8)
C12	0.0340 (10)	0.0532 (10)	0.0425 (10)	0.0036 (7)	0.0079 (8)	0.0032 (8)
C13	0.0696 (17)	0.0860 (17)	0.0429 (13)	−0.0120 (13)	0.0051 (11)	0.0021 (12)

Geometric parameters (Å, °)

F1—C13	1.290 (9)	C4—C5	1.511 (3)
F2—C13	1.327 (7)	C4—H4A	0.9700
F3—C13	1.295 (7)	C4—H4B	0.9700
F1'—C13	1.273 (7)	C5—C6	1.511 (3)
F2'—C13	1.291 (7)	C5—H5A	0.9700
F3'—C13	1.326 (10)	C5—H5B	0.9700
O1—C1	1.217 (2)	C6—H6A	0.9700
O2—C2	1.214 (2)	C6—H6B	0.9700
N1—C2	1.396 (2)	C7—C8	1.380 (3)
N1—C1	1.400 (2)	C7—C12	1.390 (2)
N1—C7	1.422 (2)	C8—C9	1.381 (3)
N2—C1	1.355 (3)	C8—H8	0.9300
N2—N3	1.406 (2)	C9—C10	1.381 (3)
N2—C3	1.453 (3)	C9—H9	0.9300
N3—C2	1.354 (3)	C10—C11	1.376 (3)
N3—C6	1.464 (3)	C10—H10	0.9300
C3—C4	1.515 (3)	C11—C12	1.387 (3)
C3—H3A	0.9700	C11—C13	1.490 (3)
C3—H3B	0.9700	C12—H12	0.9300
C2—N1—C1	109.80 (15)	C8—C7—C12	120.05 (18)
C2—N1—C7	125.31 (15)	C8—C7—N1	120.65 (16)
C1—N1—C7	124.89 (15)	C12—C7—N1	119.29 (16)
C1—N2—N3	108.32 (15)	C7—C8—C9	119.93 (19)
C1—N2—C3	125.36 (15)	C7—C8—H8	120.0
N3—N2—C3	114.04 (15)	C9—C8—H8	120.0
C2—N3—N2	109.04 (14)	C10—C9—C8	120.72 (19)
C2—N3—C6	124.67 (16)	C10—C9—H9	119.6
N2—N3—C6	113.84 (17)	C8—C9—H9	119.6
O1—C1—N2	126.13 (18)	C11—C10—C9	119.07 (19)
O1—C1—N1	127.85 (18)	C11—C10—H10	120.5
N2—C1—N1	106.00 (15)	C9—C10—H10	120.5
O2—C2—N3	126.31 (18)	C10—C11—C12	121.19 (18)
O2—C2—N1	127.89 (19)	C10—C11—C13	119.88 (18)

N3—C2—N1	105.78 (15)	C12—C11—C13	118.92 (18)
N2—C3—C4	108.52 (15)	C11—C12—C7	119.03 (17)
N2—C3—H3A	110.0	C11—C12—H12	120.5
C4—C3—H3A	110.0	C7—C12—H12	120.5
N2—C3—H3B	110.0	F1'—C13—F1	19.8 (11)
C4—C3—H3B	110.0	F1'—C13—F2'	102.7 (6)
H3A—C3—H3B	108.4	F1—C13—F2'	119.0 (9)
C5—C4—C3	111.21 (18)	F1'—C13—F3	121.8 (6)
C5—C4—H4A	109.4	F1—C13—F3	110.6 (8)
C3—C4—H4A	109.4	F2'—C13—F3	80.6 (6)
C5—C4—H4B	109.4	F1'—C13—F3'	101.7 (9)
C3—C4—H4B	109.4	F1—C13—F3'	85.2 (8)
H4A—C4—H4B	108.0	F2'—C13—F3'	108.5 (8)
C4—C5—C6	112.16 (17)	F3—C13—F3'	31.0 (10)
C4—C5—H5A	109.2	F1'—C13—F2	90.1 (7)
C6—C5—H5A	109.2	F1—C13—F2	109.1 (9)
C4—C5—H5B	109.2	F2'—C13—F2	21.3 (6)
C6—C5—H5B	109.2	F3—C13—F2	101.7 (5)
H5A—C5—H5B	107.9	F3'—C13—F2	128.5 (8)
N3—C6—C5	108.21 (15)	F1'—C13—C11	116.0 (4)
N3—C6—H6A	110.1	F1—C13—C11	113.0 (6)
C5—C6—H6A	110.1	F2'—C13—C11	115.8 (5)
N3—C6—H6B	110.1	F3—C13—C11	113.8 (5)
C5—C6—H6B	110.1	F3'—C13—C11	110.9 (6)
H6A—C6—H6B	108.4	F2—C13—C11	107.9 (4)
C1—N2—N3—C2	−10.91 (19)	C2—N3—C6—C5	166.81 (17)
C3—N2—N3—C2	−155.52 (16)	N2—N3—C6—C5	−55.5 (2)
C1—N2—N3—C6	−155.05 (16)	C4—C5—C6—N3	52.4 (2)
C3—N2—N3—C6	60.3 (2)	C2—N1—C7—C8	28.0 (3)
N3—N2—C1—O1	−172.91 (17)	C1—N1—C7—C8	−151.84 (17)
C3—N2—C1—O1	−33.3 (3)	C2—N1—C7—C12	−152.87 (16)
N3—N2—C1—N1	8.75 (18)	C1—N1—C7—C12	27.3 (2)
C3—N2—C1—N1	148.32 (18)	C12—C7—C8—C9	0.1 (3)
C2—N1—C1—O1	177.87 (19)	N1—C7—C8—C9	179.26 (16)
C7—N1—C1—O1	−2.3 (3)	C7—C8—C9—C10	−1.1 (3)
C2—N1—C1—N2	−3.83 (18)	C8—C9—C10—C11	1.1 (3)
C7—N1—C1—N2	176.00 (14)	C9—C10—C11—C12	−0.2 (3)
N2—N3—C2—O2	−173.49 (18)	C9—C10—C11—C13	−179.22 (19)
C6—N3—C2—O2	−34.1 (3)	C10—C11—C12—C7	−0.8 (3)
N2—N3—C2—N1	8.17 (19)	C13—C11—C12—C7	178.26 (18)
C6—N3—C2—N1	147.51 (17)	C8—C7—C12—C11	0.8 (3)
C1—N1—C2—O2	178.92 (19)	N1—C7—C12—C11	−178.35 (15)
C7—N1—C2—O2	−0.9 (3)	C10—C11—C13—F1	−158.8 (11)
C1—N1—C2—N3	−2.77 (18)	C12—C11—C13—F1	22.2 (12)
C7—N1—C2—N3	177.40 (15)	C10—C11—C13—F3	−31.7 (7)
C1—N2—C3—C4	165.83 (17)	C12—C11—C13—F3	149.3 (7)
N3—N2—C3—C4	−56.6 (2)	C10—C11—C13—F2	80.4 (7)
N2—C3—C4—C5	52.8 (2)	C12—C11—C13—F2	−98.6 (7)
C3—C4—C5—C6	−53.3 (2)		

Fig. 1