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(*E*)-4-Chloro-*N*′-(2,4,5-trifluorobenzylidene)benzohydrazide

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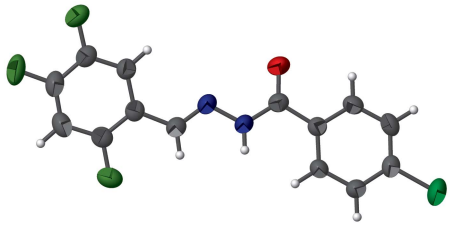
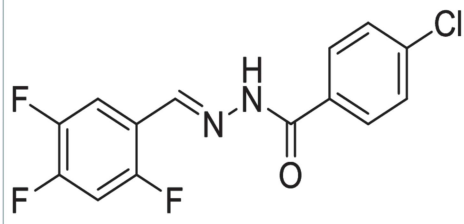
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{14}H_8ClF_3N_2O$, is approximately planar, with a dihedral angle of 4.73 (6)° between the planes of the chlorophenyl and trifluorobenzylidene rings. In the crystal, molecules are stacked in a column along the *b* axis through π–π interactions [centroid–centroid distances = 3.7097 (12) and 3.7191 (12) Å].

3D view



Chemical scheme



Structure description

Hydrazones have continued to attract interest from researchers due to their medicinal applications. These derivatives exhibit antimicrobial (Pieczonka *et al.*, 2013), antiproliferative (Yadagiri *et al.*, 2014), antiplatelet (Mashayekhi *et al.*, 2013), anti-hepatitis (Şenkardeş *et al.*, 2016) and anti-amoebic (Siddiqui *et al.*, 2012) activities.

The geometric parameters of the title molecule (Fig. 1) agree well with those reported for similar structures (Sreeja *et al.*, 2013; Nair *et al.*, 2012). The chlorophenyl ring makes a dihedral angle of 4.73 (6)° with the trifluorobenzylidene ring. In the crystal, the molecules are linked by π–π interactions [$Cg1\cdots Cg2^{i}$ = 3. 7191 (12) Å and $Cg1\cdots Cg2^{ii}$ = 3. 7096 (12) Å; symmetry codes: (i) $1-x, -y, 2-z$; (ii) $1-x, 1-y, 2-z$]; $Cg1$ and $Cg2$ are the centroids of the C1–C6 and C9–C14 rings, respectively;

Synthesis and crystallization

To an ethanolic solution of 4-chlorobenzohydrazide (0.17 g, 0.001 mol), 2,4,5-trifluoro benzaldehyde (0.1 mL, 0.001 mol) and a few drops of conc. HCl were added. The reaction mixture was stirred well at room temperature for 30 min. The insoluble solid gradually

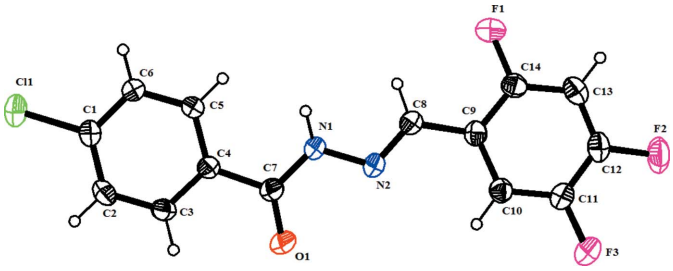


Figure 1
The molecular structure of the title compound, with atom labels and 30% probability displacement ellipsoids for non-H atoms.

generated was filtered and washed with petroleum benzine (60–80°C) and dried in a vacuum desiccator. The crude solid was recrystallized from DMSO solution (yield 98%).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_8ClF_3N_2O$
M_r	312.67
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	295
a, b, c (Å)	6.4998 (2), 7.4286 (2), 14.8289 (4)
α, β, γ (°)	84.859 (2), 86.707 (2), 83.313 (2)
V (Å^3)	707.47 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.30
Crystal size (mm)	0.18 × 0.16 × 0.11
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 1996)
T_{min}, T_{max}	0.948, 0.968
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10440, 2925, 2162
R_{int}	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.628
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.120, 0.99
No. of reflections	2925
No. of parameters	191
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.18

Computer programs: *APEX2* (Bruker, 2008), *SAINT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008), *PLATON* (Spek, 2009).

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full crystallographic data

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Crystal data

$C_{14}H_8ClF_3N_2O$
M_r = 312.67
Triclinic, $P\overline{1}$
a = 6.4998 (2) Å
b = 7.4286 (2) Å
c = 14.8289 (4) Å
α = 84.859 (2)°
β = 86.707 (2)°
γ = 83.313 (2)°
V = 707.47 (3) Å^3
Z = 2
F(000) = 316
D_x = 1.468 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2925 reflections
θ = 1.4–26.5°
μ = 0.30 mm^{-1}
T = 295 K
Block, colourless
0.18 × 0.16 × 0.11 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.948, T_{max} = 0.968
10440 measured reflections
2925 independent reflections
2162 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 26.5°, θ_{min} = 1.4°
h = −8→8
k = −9→9
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.120
S = 0.99
2925 reflections
191 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
$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.2152P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.18109 (10)	0.17309 (9)	0.53712 (4)	0.0773 (2)
F1	0.21764 (19)	0.46649 (19)	1.23486 (9)	0.0731 (4)

F2	0.7236 (3)	0.3478 (2)	1.45102 (8)	0.0871 (5)
N2	0.5848 (2)	0.2425 (2)	1.03868 (10)	0.0488 (4)
N1	0.4890 (2)	0.2335 (2)	0.95876 (10)	0.0489 (4)
H1	0.3580	0.2656	0.9555	0.059*
F3	0.9897 (2)	0.19270 (19)	1.32932 (8)	0.0738 (4)
C4	0.4866 (3)	0.1731 (2)	0.80147 (11)	0.0429 (4)
C5	0.2782 (3)	0.2351 (3)	0.79365 (12)	0.0479 (4)
H5	0.2005	0.2772	0.8437	0.058*
C8	0.4666 (3)	0.3037 (3)	1.10238 (13)	0.0497 (4)
H8	0.3278	0.3410	1.0918	0.060*
C2	0.5077 (3)	0.1097 (3)	0.64454 (13)	0.0542 (5)
H2	0.5842	0.0676	0.5942	0.065*
C7	0.6013 (3)	0.1743 (3)	0.88617 (13)	0.0494 (4)
C13	0.4658 (4)	0.4083 (3)	1.34516 (13)	0.0578 (5)
H13	0.3728	0.4619	1.3876	0.069*
C6	0.1850 (3)	0.2352 (3)	0.71227 (12)	0.0500 (5)
H6	0.0453	0.2771	0.7073	0.060*
O1	0.7870 (2)	0.1260 (3)	0.88799 (10)	0.0826 (5)
C1	0.3014 (3)	0.1725 (3)	0.63871 (12)	0.0499 (5)
C9	0.5423 (3)	0.3175 (2)	1.19213 (12)	0.0455 (4)
C14	0.4111 (3)	0.3957 (3)	1.25786 (13)	0.0511 (5)
C10	0.7425 (3)	0.2490 (3)	1.21677 (12)	0.0483 (4)
H10	0.8368	0.1959	1.1747	0.058*
C3	0.5999 (3)	0.1097 (3)	0.72598 (13)	0.0492 (4)
H3	0.7396	0.0670	0.7305	0.059*
C11	0.7985 (3)	0.2609 (3)	1.30357 (13)	0.0519 (5)
C12	0.6613 (4)	0.3393 (3)	1.36670 (13)	0.0574 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0880 (5)	0.1003 (5)	0.0470 (3)	−0.0101 (3)	−0.0175 (3)	−0.0158 (3)
F1	0.0537 (7)	0.0842 (9)	0.0778 (8)	0.0098 (6)	0.0005 (6)	−0.0118 (7)
F2	0.1174 (12)	0.1016 (11)	0.0447 (6)	−0.0074 (9)	−0.0183 (7)	−0.0162 (7)
N2	0.0461 (9)	0.0574 (10)	0.0442 (8)	−0.0059 (7)	−0.0099 (7)	−0.0063 (7)
N1	0.0406 (8)	0.0628 (10)	0.0442 (8)	−0.0019 (7)	−0.0091 (6)	−0.0106 (7)
F3	0.0619 (8)	0.0937 (10)	0.0658 (7)	−0.0006 (7)	−0.0223 (6)	−0.0040 (7)
C4	0.0417 (10)	0.0439 (10)	0.0433 (9)	−0.0049 (7)	−0.0022 (7)	−0.0047 (7)
C5	0.0405 (10)	0.0593 (11)	0.0436 (9)	0.0005 (8)	0.0005 (7)	−0.0117 (8)
C8	0.0459 (10)	0.0553 (11)	0.0490 (10)	−0.0060 (8)	−0.0069 (8)	−0.0070 (8)
C2	0.0591 (12)	0.0582 (12)	0.0456 (10)	−0.0056 (9)	0.0084 (9)	−0.0140 (9)
C7	0.0414 (10)	0.0577 (11)	0.0488 (10)	−0.0044 (8)	−0.0040 (8)	−0.0041 (8)
C13	0.0728 (14)	0.0519 (12)	0.0480 (10)	−0.0064 (10)	0.0095 (10)	−0.0092 (9)
C6	0.0412 (10)	0.0596 (12)	0.0494 (10)	0.0000 (8)	−0.0062 (8)	−0.0098 (8)
O1	0.0419 (8)	0.1419 (16)	0.0626 (9)	0.0102 (9)	−0.0110 (7)	−0.0215 (9)
C1	0.0579 (12)	0.0519 (11)	0.0417 (9)	−0.0089 (9)	−0.0056 (8)	−0.0082 (8)
C9	0.0501 (10)	0.0434 (10)	0.0444 (9)	−0.0106 (8)	−0.0039 (8)	−0.0039 (8)
C14	0.0507 (11)	0.0472 (10)	0.0549 (11)	−0.0041 (8)	0.0001 (9)	−0.0045 (8)

C10	0.0502 (11)	0.0502 (11)	0.0455 (9)	−0.0073 (8)	−0.0032 (8)	−0.0065 (8)
C3	0.0414 (10)	0.0524 (11)	0.0533 (10)	−0.0012 (8)	0.0014 (8)	−0.0092 (8)
C11	0.0562 (12)	0.0507 (11)	0.0497 (10)	−0.0081 (9)	−0.0106 (9)	−0.0007 (8)
C12	0.0786 (15)	0.0541 (12)	0.0417 (10)	−0.0123 (10)	−0.0078 (9)	−0.0064 (8)

Geometric parameters (Å, º)

Cl1—C1	1.7363 (18)	C2—C1	1.372 (3)
F1—C14	1.356 (2)	C2—C3	1.379 (3)
F2—C12	1.345 (2)	C2—H2	0.9300
N2—C8	1.266 (2)	C7—O1	1.220 (2)
N2—N1	1.380 (2)	C13—C12	1.358 (3)
N1—C7	1.343 (2)	C13—C14	1.376 (3)
N1—H1	0.8600	C13—H13	0.9300
F3—C11	1.349 (2)	C6—C1	1.376 (3)
C4—C5	1.387 (3)	C6—H6	0.9300
C4—C3	1.391 (2)	C9—C14	1.380 (3)
C4—C7	1.498 (2)	C9—C10	1.397 (3)
C5—C6	1.381 (2)	C10—C11	1.371 (2)
C5—H5	0.9300	C10—H10	0.9300
C8—C9	1.461 (2)	C3—H3	0.9300
C8—H8	0.9300	C11—C12	1.375 (3)
C8—N2—N1	114.85 (15)	C1—C6—H6	120.4
C7—N1—N2	119.69 (15)	C5—C6—H6	120.4
C7—N1—H1	120.2	C2—C1—C6	121.46 (17)
N2—N1—H1	120.2	C2—C1—Cl1	120.08 (15)
C5—C4—C3	118.83 (16)	C6—C1—Cl1	118.46 (15)
C5—C4—C7	124.05 (16)	C14—C9—C10	117.17 (17)
C3—C4—C7	117.10 (16)	C14—C9—C8	119.73 (17)
C6—C5—C4	120.65 (16)	C10—C9—C8	123.07 (17)
C6—C5—H5	119.7	F1—C14—C13	117.91 (17)
C4—C5—H5	119.7	F1—C14—C9	118.15 (17)
N2—C8—C9	121.75 (17)	C13—C14—C9	123.93 (18)
N2—C8—H8	119.1	C11—C10—C9	119.38 (18)
C9—C8—H8	119.1	C11—C10—H10	120.3
C1—C2—C3	119.12 (17)	C9—C10—H10	120.3
C1—C2—H2	120.4	C2—C3—C4	120.77 (17)
C3—C2—H2	120.4	C2—C3—H3	119.6
O1—C7—N1	122.38 (17)	C4—C3—H3	119.6
O1—C7—C4	121.28 (17)	F3—C11—C10	120.38 (18)
N1—C7—C4	116.33 (16)	F3—C11—C12	118.47 (17)
C12—C13—C14	117.17 (18)	C10—C11—C12	121.14 (19)
C12—C13—H13	121.4	F2—C12—C13	119.85 (19)
C14—C13—H13	121.4	F2—C12—C11	118.9 (2)
C1—C6—C5	119.17 (17)	C13—C12—C11	121.21 (18)
C8—N2—N1—C7	178.48 (18)	C12—C13—C14—C9	0.1 (3)

C3—C4—C5—C6	−0.4 (3)	C10—C9—C14—F1	178.60 (17)
C7—C4—C5—C6	177.82 (18)	C8—C9—C14—F1	−3.5 (3)
N1—N2—C8—C9	178.32 (16)	C10—C9—C14—C13	−0.5 (3)
N2—N1—C7—O1	0.6 (3)	C8—C9—C14—C13	177.41 (19)
N2—N1—C7—C4	−178.52 (15)	C14—C9—C10—C11	0.5 (3)
C5—C4—C7—O1	−176.5 (2)	C8—C9—C10—C11	−177.34 (17)
C3—C4—C7—O1	1.8 (3)	C1—C2—C3—C4	−0.2 (3)
C5—C4—C7—N1	2.7 (3)	C5—C4—C3—C2	0.5 (3)
C3—C4—C7—N1	−179.10 (17)	C7—C4—C3—C2	−177.88 (18)
C4—C5—C6—C1	0.1 (3)	C9—C10—C11—F3	178.76 (17)
C3—C2—C1—C6	−0.1 (3)	C9—C10—C11—C12	−0.1 (3)
C3—C2—C1—Cl1	−179.75 (15)	C14—C13—C12—F2	180.00 (18)
C5—C6—C1—C2	0.2 (3)	C14—C13—C12—C11	0.3 (3)
C5—C6—C1—Cl1	179.82 (15)	F3—C11—C12—F2	1.1 (3)
N2—C8—C9—C14	175.94 (18)	C10—C11—C12—F2	180.00 (18)
N2—C8—C9—C10	−6.3 (3)	F3—C11—C12—C13	−179.17 (19)
C12—C13—C14—F1	−178.97 (18)	C10—C11—C12—C13	−0.3 (3)