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N-[4-Cyano-3-(trifluoromethyl)phenyl]-3-(4-fluorophenylsulfanyl)-2-hydroxy-2-methylpropionamide

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.065
wR factor = 0.175
Data-to-parameter ratio = 16.3

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

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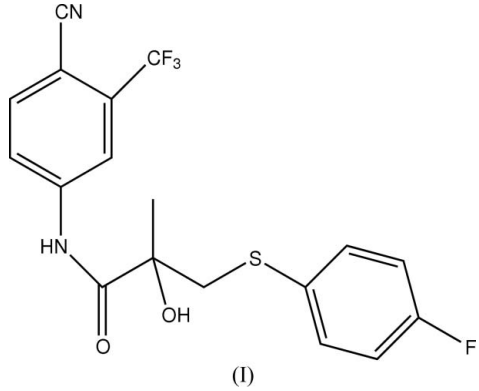
The title compound, $C_{18}H_{14}F_4N_2O_2S$, crystallizes in a conformation where the two benzene rings are parallel. In the crystal structure, molecules form one-dimensional extended chains *via* O—H···N hydrogen bonds.

Comment

The title compound, (I), was prepared as an intermediate in the synthesis of bicalutamide, a nonsteroidal antiandrogen which acts on the male hormones (androgens) but has no steroidal effects (Tucker *et al.*, 1988; Xiao *et al.*, 2003). The crystal structure of *N*-(4-cyano-3-trifluoromethyl-phenyl)-3-(4-fluorophenylsulfanyl)-2-hydroxy-2-methylpropionamide chloroform solvate has been reported (Tang & Gu, 2005). We report here the crystal structure of solvent-free *N*-[4-cyano-3-(trifluoromethyl)phenyl]-3-(4-fluorophenylsulfanyl)-2-hydroxy-2-methylpropionamide, (I). In the crystal structure, there are minor differences in the conformation compared with that of the chloroform solvate. The bond lengths and bond angles are identical. The two aromatic ring planes are parallel to each other, forming a dihedral angle of 1.22 (18)°. Molecules are linked into one-dimensional extended chains by intermolecular hydrogen bonds (Table 1).



(I)

Experimental

2-Hydroxy-2-methyl-3-*p*-tolylsulfanylpropionic acid (36.5 g, 159 mmol), 2-trifluoromethyl-4-aminobenzonitrile (38.0 g, 200 mmol) and *N*,*N*-dimethylacetamide (300 ml) were mixed in a 500 ml three-necked bottle, and stirred at 263 K. Thionyl chloride (12 ml, 164 mmol) was then dropped into the above solution over a period of 30 min, with the reaction mixture kept at 258–263 K; the mixture continued to react for 24 h. After that time, the reaction solution was poured into 600 ml of ice–water, followed by extraction with CH_2Cl_2. The organic phase was washed with HCl (3 mol l^{-1}, 4 × 300 ml), NaCl (saturated solution, 2 × 150 ml), $NaHCO_3$ (saturated solution, 3 × 150 ml) and NaCl (saturated solution, 2 × 150 ml), respectively, and then dried with anhydrous $MgSO_4$; the CH_2Cl_2 was removed

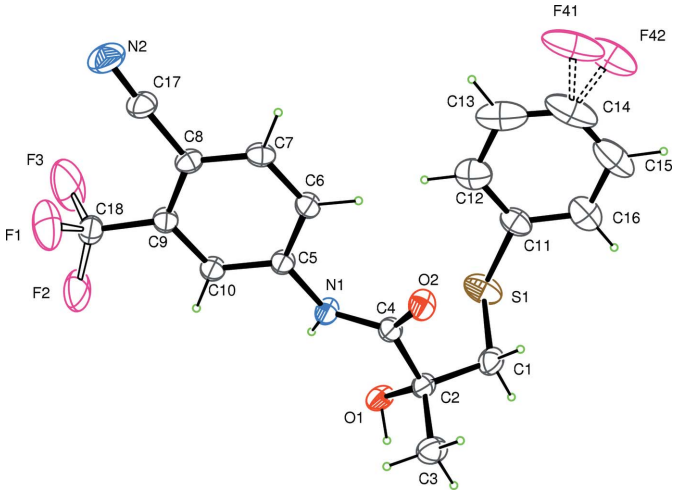


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. Both disorder components are shown.

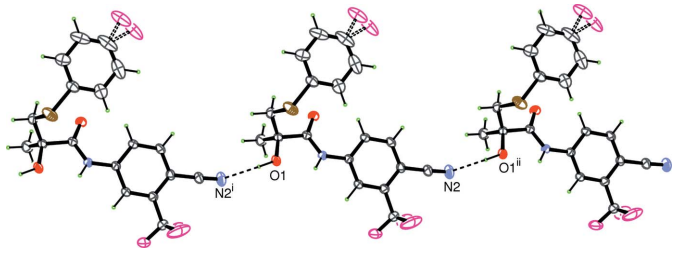


Figure 2
A chain of molecules in (I). Displacement ellipsoids are drawn at the 30% probability level and hydrogen bonds are shown as dashed lines. Both disorder components are shown. [Symmetry codes: (i) $x-1, y-1, z-1$ (ii) $x+1, y+1, z+1$.]

under vacuum distillation. The final product was recrystallized from a mixture of toluene and petroleum ether (5:1 *v*/*v*), and crystals (26 g, yield 41%) were obtained (Sepp-Lorenzino & Slovin, 2000; Thurlow, 1998). Colorless crystals of (I) suitable for X-ray diffraction were obtained from acetone.

Crystal data

$C_{18}H_{14}F_4N_2O_2S$
$M_r = 398.37$
Triclinic, $P\overline{1}$
$a = 10.136\ (5)$ Å
$b = 10.343\ (6)$ Å
$c = 10.524\ (4)$ Å
$\alpha = 106.82\ (2)^\circ$
$\beta = 116.97\ (2)^\circ$
$\gamma = 95.19\ (2)^\circ$
$V = 908.9\ (8)$ Å^3
$Z = 2$
$D_x = 1.456$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.23$ mm^{-1}
$T = 296\ (1)$ K
Block, colorless
0.28 × 0.25 × 0.17 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: none
9009 measured reflections
4135 independent reflections
2814 reflections with $F^2 > 2\sigma(F^2)$
$R_{int} = 0.026$
$\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.175$
$S = 1.01$
4135 reflections
253 parameters
H-atom parameters constrained
$w = 1/[0.0015F_o^2 + 3\sigma(F_o^2) + 0.5]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H101···N2^{i}	0.92	2.00	2.918 (2)	178
N1—H111···O1^{ii}	0.86	2.62	3.167 (3)	123

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

H atoms of the hydroxy and amide groups were located in difference Fourier maps and included in the refinement as riding, with the as-found O—H and N—H bond lengths; their isotropic displacement parameters were initially refined and then fixed in the final stage. The other H atoms were placed in calculated positions with C—H = 0.96–0.98 Å and included in the refinement in a riding model, with $U_{iso}(H) = 1.2U_{eq}$(carrier atom).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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