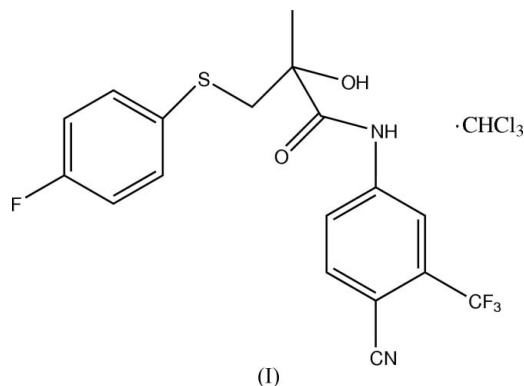


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Key indicators

Single-crystal X-ray study
T = 253 K
Mean σ (C–C) = 0.003 Å
Disorder in main residue
R factor = 0.047
wR factor = 0.089
Data-to-parameter ratio = 14.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.***N*-[4-Cyano-3-(trifluoromethyl)phenyl]-3-(4-fluorophenylsulfanyl)-2-hydroxy-2-methylpropionamide chloroform solvate**The title compound, C₁₈H₁₄F₄N₂O₂S·CHCl₃, forms a one-dimensional hydrogen-bonded chain *via* a single O—H···N interaction; the chain runs approximately along the [110] axis. The two benzene rings are almost parallel to one another, forming a dihedral angle of 8.43 (8)°.

Comment

The title compound, (I), was prepared as an intermediate in the synthesis of bicalutamide [trademark name for *N*-(4-cyano-3-trifluoromethylphenyl)-3-(4-fluorobenzensulfonyl)-2-hydroxy-2-methylpropionamide], a nonsteroidal anti-androgen which acts on the male hormones (androgens) but which has no steroidal effects (Tucker *et al.*, 1988; Xiao *et al.*, 2003).The crystal structure of (I) comprises one *N*-(4-cyano-3-trifluoromethylphenyl)-3-(4-fluorophenylsulfanyl)-2-hydroxy-2-methylpropionamide molecule and one CHCl₃ solvent molecule. The solvent molecule is disordered. The two benzene ring mean planes are almost parallel to each other, forming a dihedral angle of 8.43 (8)°.Intermolecular O—H···N hydrogen bonds (Table 2) link the molecules into infinite chains, parallel to the [110] axis. Atom O1 belonging to the hydroxy group acts a donor, while N2 atom of the cyano group serves as an acceptor. Because of this hydrogen-bond interaction, the cyano group deviates slightly from the plane of the benzene ring (C5–C8/C10/C12); the deviations of atoms C9 and N2 from the mean plane are 0.123 and 0.250 Å, respectively. The cyano group is unconjugated with the benzene ring, resulting in a C8–C9 bond length of 1.448 (3) Å, which is long compared with the value expected for a formal *C*sp²–*C*sp bond (Allen *et al.*, 1987). The chain is further stabilized by weak intermolecular N1–H1···O1 interactions (Table 2) to form layers in the crystal structure.Received 1 August 2005
Accepted 5 September 2005
Online 14 September 2005

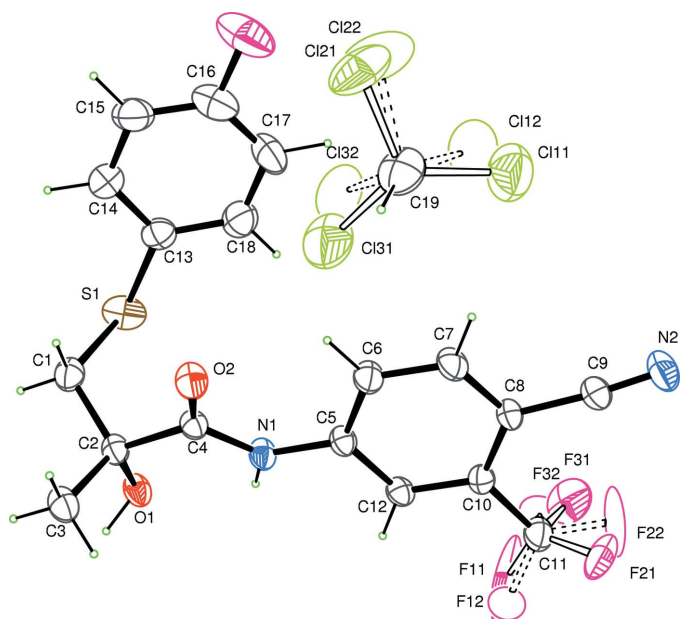


Figure 1
A view of (I), with displacement ellipsoids drawn at the 30% probability level and H atoms shown as small circles of arbitrary radii. The minor component disordered Cl atoms in the solvent and F atoms in the CF₃ group are depicted with unshaded ellipsoids and dashed bonds.

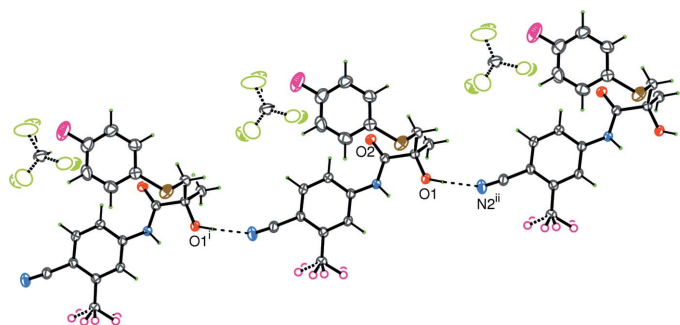


Figure 2
A chain of molecules in (I). Hydrogen bonds are represented with dashed lines. [Symmetry codes: (i) $1 + x, 1 + y, z$; (ii) $-1 + x, -1 + y, z$.]

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*-dimethyl acetamide (300 ml) were mixed in a 500 ml three-necked bottle and stirred at 263 K. Thionyl chloride (12 ml, 164 mmol) was then added dropwise to the solution and the resulting mixture was kept for 30 min at 258–263 K and then allowed to react for 24 h. After that time, the reaction solution was poured into ice-water (600 ml) and extracted with CH₂Cl₂ (4 × 300 ml). The organic part was washed with HCl (3 M, 4 × 300 ml), NaCl (saturated solution, 2 × 150 ml), NaHCO₃ (saturated solution, 3 × 150 ml) and NaCl (saturated solution, 2 × 150 ml), and dried over anhydrous MgSO₄. Compound (I) was recovered after vacuum distillation. The final product was recrystallized from a mixture of toluene and petroleum ether (5:1) (yield 26 g, 41%) (Thurlow, 1998; Sepp-Lorenzino & Slovin, 2000). Finally, compound (I) was recrystallized from chloroform, giving colourless crystals suitable for X-ray diffraction.

Crystal data

C₁₈H₁₄F₄N₂O₂S·CHCl₃
M_r = 517.75
 Triclinic, *P* $\bar{1}$
a = 8.8442 (9) Å
b = 10.4591 (9) Å
c = 13.319 (1) Å
 α = 110.718 (4)°
 β = 90.895 (2)°
 γ = 105.595 (2)°
V = 1101.5 (2) Å³

Z = 2
D_x = 1.561 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 4876 reflections
 θ = 2.2–27.5°
 μ = 0.56 mm⁻¹
T = 253 (1) K
 Block, colourless
 0.42 × 0.33 × 0.28 mm

Data collection

Rigaku R-Axis RAPID
 diffractometer
 ω scans
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
T_{min} = 0.738, *T_{max}* = 0.854
 9744 measured reflections

4896 independent reflections
 3260 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.025
 θ_{max} = 27.5°
 h = -11 → 11
 k = -13 → 13
 l = -17 → 17

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.047
 $wR(F^2)$ = 0.089
 S = 0.96
 4892 reflections
 334 parameters

H-atom parameters constrained
 $w = 1/[0.0002F_o^2 + 3\sigma(F_o^2) + 0.5]$
 (ΔF_o^2)
 $(\Delta/\sigma)_{max}$ = 0.010
 $\Delta\rho_{max}$ = 0.39 e Å⁻³
 $\Delta\rho_{min}$ = -0.38 e Å⁻³

Table 1

Selected bond lengths (Å).

| | | | |
|--------|-----------|-------|-----------|
| S1–C1 | 1.801 (3) | O2–C4 | 1.218 (2) |
| S1–C13 | 1.769 (2) | N2–C9 | 1.137 (3) |
| O1–C2 | 1.425 (2) | C8–C9 | 1.448 (3) |

Table 2

Hydrogen-bond geometry (Å, °).

| <i>D</i> –H··· <i>A</i> | <i>D</i> –H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> –H··· <i>A</i> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| O1–H101···N2 ⁱ | 0.90 | 2.06 | 2.957 (3) | 175 |
| N1–H111···O1 ⁱⁱ | 0.86 | 2.51 | 3.094 (3) | 126 |

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

The Cl atoms of the chloroform molecule and the F atoms of the CF₃ group were found to be disordered. Each atom was split over two sites and the site occupancy factors were refined with the sums constrained to 1, and then fixed at the final stage. H atoms belonging to the hydroxy and amido groups and to the CHCl₃ molecule were located in difference maps and included in the refinement with as-found constrained O–H, N–H and C–H bond lengths. Other H atoms were placed in calculated positions and refined using a riding model. Constrained C–H bond lengths are 0.98 Å for aromatic CH, 0.97 Å for methylene CH₂ and 0.96 Å for methyl CH₃. For all H atoms, *U*_{iso}(H) values were initially refined and then fixed in the final cycles.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); 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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