

Li-Rong Wen,^a Wei-Si Guo,^a
Ming Li,^a Xue-Mei Li,^a Shu-Sheng
Zhang,^{a*} Ping-Kai Ouyang^b and
Zi-Qin Ke^c

^aCollege of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, 266042 Qingdao, Shandong, People's Republic of China, ^bCollege of Life Science and Pharmaceutical Engineering, Nanjing University of Technology, 210093 Nanjing, Jiangsu, People's Republic of China, and ^cDepartment of Chemistry, Xinjiang Normal University, 830054 Urumuqi, Xinjiang, People's Republic of China

Correspondence e-mail:
zhangshush@public.qd.sd.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$
Disorder in main residue
R factor = 0.061
wR factor = 0.189
Data-to-parameter ratio = 11.6

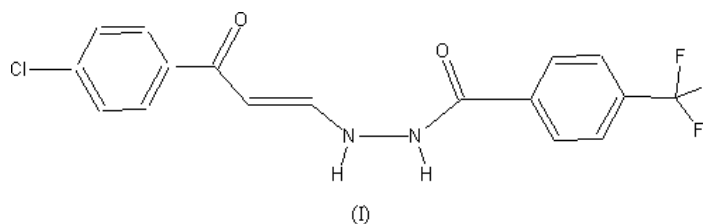
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

1-(4-Chlorophenyl)-3-(4-trifluoromethylbenzoyl-hydrazino)-2-propenone

The title compound, $\text{C}_{17}\text{H}_{12}\text{ClF}_3\text{N}_2\text{O}_2$, contains two independent molecules in the asymmetric unit. The dihedral angles between the two benzene rings in each molecule are $53.9(2)$ and $23.1(2)^\circ$. The crystal packing is stabilized by intermolecular hydrogen bonds, which link the molecules into two-dimensional layers.

Comment

In recent years, it has been reported that enamines are important and versatile reagents, which have been extensively used as building blocks in organic synthesis (Olivera *et al.*, 2000; Hernandez *et al.*, 2003). The introduction of the trifluoromethyl group into molecules has become increasingly significant, because the incorporation of this functional group has frequently generated much more activity than that of the parent compounds (Welch, 1987; Lipshutz, 1986; Cao *et al.*, 2002). In this paper, we report the synthesis and crystal structure of the title compound, (I).



The structure of (I) consists of two crystallographically independent molecules *A* and *B* in the asymmetric unit of the centrosymmetric space group $P\bar{1}$ (Fig. 1). The bond lengths and angles in *A* and *B* (Table 1) agree with each other and are within normal ranges (Allen *et al.*, 1987). All the bond lengths except for those involving the halogen atoms show a character intermediate between single and double bonds because of π -conjugation effects in the molecules. Both the molecules are non-planar, with dihedral angles of $53.9(2)$ and $23.1(2)^\circ$ between the two benzene planes in *A* and *B*, respectively. In molecule *A*, the three F atoms show positional disorder, with refined site occupancies of 0.53 (3) and 0.47 (3) for the major and minor components, respectively.

There exists one intramolecular $\text{N}-\text{H}\cdots\text{O}$ interaction in each molecule (Table 2), forming a six-membered ring. In the crystal structure, molecules are linked into chains by intermolecular $\text{N}1-\text{H}1\text{A}\cdots\text{O}3$ and $\text{N}3-\text{H}3\text{A}\cdots\text{O}1$ hydrogen bonds. Two other intermolecular interactions (Table 2) connect the chains into two-dimensional layers (Fig. 2).

Received 5 November 2004

Accepted 19 November 2004

Online 27 November 2004

Experimental

The title compound was prepared by the reaction of 1-(4-chlorophenyl)-3-(dimethylamino)-2-propenone (0.625 g, 3 mmol) and 4-(trifluoromethyl)benzoylhydrazine (0.61 g, 3 mmol) in glacial acetic acid (15 ml), with stirring for 2 h at room temperature. The solution was filtered and single crystals suitable for an X-ray diffraction study were obtained from a mixture of EtOH/DMF (3:1 v/v).

Crystal data

$C_{17}H_{12}ClF_3N_2O_2$	$Z = 4$
$M_r = 368.74$	$D_x = 1.504 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.6851 (14) \text{ \AA}$	Cell parameters from 8493 reflections
$b = 8.8523 (16) \text{ \AA}$	$\theta = 0.8\text{--}25.0^\circ$
$c = 25.104 (4) \text{ \AA}$	$\mu = 0.28 \text{ mm}^{-1}$
$\alpha = 86.897 (3)^\circ$	$T = 293 (2) \text{ K}$
$\beta = 88.293 (3)^\circ$	Column, colorless
$\gamma = 72.739 (3)^\circ$	$0.32 \times 0.24 \times 0.16 \text{ mm}$
$V = 1628.4 (5) \text{ \AA}^3$	

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	5711 independent reflections
ω scans	3546 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.021$
$T_{\text{min}} = 0.916$, $T_{\text{max}} = 0.957$	$\theta_{\text{max}} = 25.0^\circ$
8493 measured reflections	$h = -4 \rightarrow 9$
	$k = -10 \rightarrow 10$
	$l = -29 \rightarrow 29$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0816P)^2 + 0.8468P]$
$R[F^2 > 2\sigma(F^2)] = 0.061$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.189$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.09$	$\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$
5711 reflections	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
494 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Selected bond lengths (\AA).

Cl1—C15	1.733 (4)	C8—F2	1.319 (7)
Cl2—C32	1.742 (4)	C25—F4	1.307 (4)
C8—F1	1.280 (7)	C25—F6	1.318 (4)
C8—F3	1.300 (7)	C25—F5	1.327 (4)
C8—F3'	1.300 (7)	N1—N2	1.385 (4)
C8—F2'	1.315 (7)	N3—N4	1.374 (4)
C8—F1'	1.319 (7)		

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
N1—H1A \cdots O3 ⁱ	0.86 (3)	1.98 (3)	2.817 (4)	165 (2)
N2—H2A \cdots O2	0.86 (3)	1.99 (3)	2.663 (5)	135 (3)
N3—H3A \cdots O1 ⁱⁱ	0.86 (2)	2.03 (2)	2.844 (4)	158 (2)
N4—H4A \cdots O4	0.86 (2)	1.81 (2)	2.560 (4)	145 (3)
C2—H2 \cdots F3 ⁱⁱⁱ	0.93	2.42	3.218 (1)	142
C22—H22 \cdots O3 ⁱ	0.93	2.53	3.398 (5)	156

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

All the H atoms were positioned geometrically and treated as riding. H atoms on N atoms were refined isotropically. All other H atoms were refined with C—H distances of 0.93 \AA and with $U_{\text{iso}}(\text{H}) =$

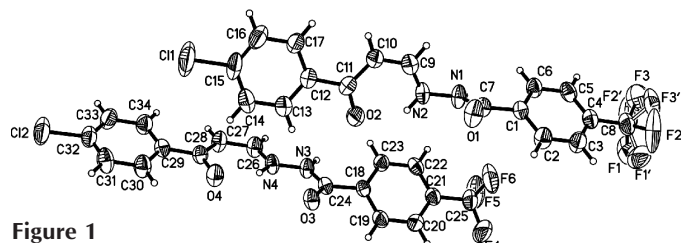


Figure 1

View of the asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Both disorder components are shown.

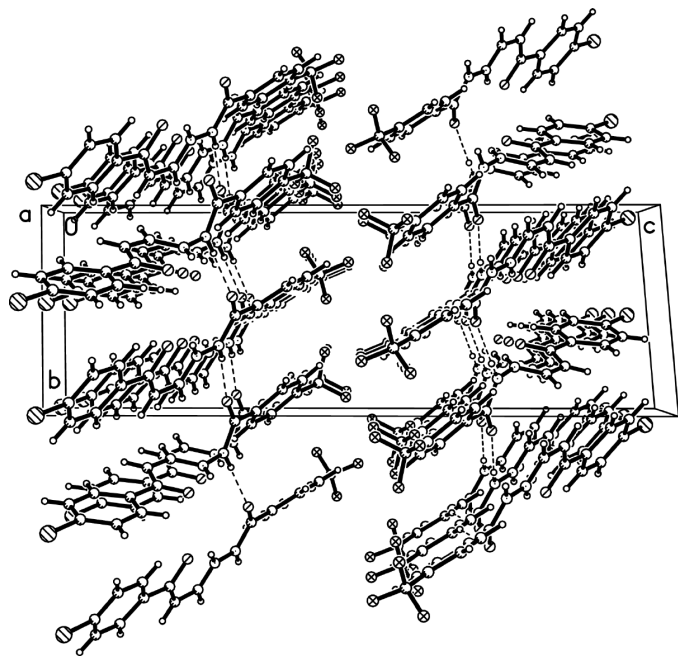


Figure 2

Packing diagram of (I). Dashed lines denote short-contact C—H \cdots O interactions. Only the major component of the disordered CF_3 group is drawn.

$1.2U_{\text{eq}}(\text{C})$. Owing to the large fraction of weak data at higher angles, 2θ maximum was 50° .

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 1997); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON (Spek, 2003).

This project was supported by the Natural Science Foundation of Shandong Province (No. Y2003B01) and the Outstanding Adult-Young Scientific Research Encouraging Foundation of Shandong Province (No. 03BS081).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Cao, S. C., Qian, X. H., Song, G. H. & Huang, Q. C. (2002). *J. Fluorine Chem.* **117**, 63–66.
- Hernandez, S., Sanmantine, R., Tellitu, I. & Dominguez, E. (2003). *Org. Lett.* **5**, 1095–1098.

- Lipshutz, B. H. (1986). *Chem. Rev.* **86**, 795–819.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Olivera, R., Sanmartin, R., Tellitu, I. & Dominguez, E. (2000). *Tetrahedron Lett.* **41**, 4353–4356.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
- Siemens (1996). *SMART* and *SAINTE*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Welch, T. T. (1987). *Tetrahedron*, **43**, 3123–3197.