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3,4,4-Trichloro-1-(4-chlorophenylsulfanyl)-1-[4-(2-fluorophenyl)piperazinyl]-2-nitrobuta-1,3-diene

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Key indicators

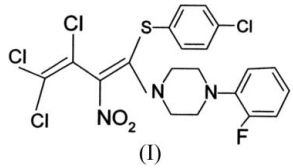
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.118
Data-to-parameter ratio = 21.4

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

In the title *N*,*S*-substituted compound, $C_{20}H_{16}Cl_4FN_3O_2S$, the piperazine ring adopts a chair conformation. The butadiene unit assumes a configuration close to *cisoid*.

Comment

Substituted piperazine compounds are important for clinical chemistry (Soladin & Heat, 1996) and gene transfer reactions (Zhao & Miller, 1996). Some piperazine derivatives possess high biological activity for multidrug resistance in cancer (Suzuki *et al.*, 1997) and malaria (Takayamagi *et al.*, 2003). *N*,*S*-Substituted 1,3-halodiene compounds have been prepared from the reaction of some mono(thio)substituted dienes with some amines [primary amine, heterocyclic amine (piperazine, morpholine, *etc.*)] (Ibis & Gokmen, 1998). The title compound, (I), was synthesized from the reaction of 1,3,4,4-tetrachloro-1-(4-chlorophenylsulfanyl)-2-nitro-1,3-butadiene with 1-(2-fluorophenylpiperazine (Ibis *et al.*, 2004). The piperazine ring is in a chair conformation. The C—C bond lengths of the butadiene unit are similar to those in related compounds (Surange *et al.*, 1997; Ibis *et al.*, 2006, 2006*a*,*b*). The C4—C3—C2—C1 torsion angle is 69.0 (3)°, a value consistent with corresponding values in similar compounds (Verenich *et al.*, 1992; Surange *et al.*, 1997; Ibis *et al.*, 2006, 2006*a*,*b*).



(I)

Experimental

1,3,4,4-Tetrachloro-1-(4-chlorophenylsulfanyl)-2-nitro-1,3-butadiene (0.1 g, 0.263 mmol) and 1-(2-fluorophenyl)piperazine (0.079 g, 0.263 mmol) were mixed in dry diethyl ether (25 ml). The mixture was stirred at room temperature for 2–3 h. After completion of the reaction, the organic layer was extracted with $CHCl_3$, washed with water (4 × 30 ml) and dried with anhydrous $MgSO_4$. The organic extract was concentrated on a rotary evaporator and the yellow crystalline product, (I), was obtained. Yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution (yield 0.104 g, 76%; m.p. 461–462 K).

Crystal data

$C_{20}H_{16}Cl_4FN_3O_2S$
M_r = 523.24
Monoclinic, $P2_1/n$
a = 13.7862 (4) Å
b = 11.0663 (3) Å
c = 14.6971 (4) Å
β = 93.277 (1)°
V = 2238.56 (11) Å^3
Z = 4
D_x = 1.552 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.65 mm^{-1}
T = 293.5 K
Prism, yellow
0.50 × 0.40 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID S diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.682, T_{max} = 0.878
131970 measured reflections
6892 independent reflections
6335 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.035
θ_{max} = 30.1°

Refinement

Refinement on F
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.118
S = 1.09
6335 reflections
280 parameters
All H-atom parameters constrained
Chebychev polynomial with 3 parameters (Carruthers & Watkin, 1979) 5.3958, −5.3010, 2.8963
$(\Delta/\sigma)_{max}$ = 0.006
$\Delta\rho_{max}$ = 0.93 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C5	1.781 (2)	C3—C2	1.454 (3)
C3—C4	1.387 (3)	C1—C2	1.314 (3)
C4—C3—C2	124.9 (2)	C3—C2—C1	125.2 (2)
C4—S1—C5—C6	56.7 (2)	N3—C12—C11—N2	58.1 (2)
C5—S1—C4—N2	37.5 (2)	N3—C13—C14—N2	−55.0 (2)
C13—N3—C15—C16	170.6 (2)	N1—C3—C2—Cl3	65.4 (2)
C4—N2—C14—C13	−130.2 (2)	C4—C3—C2—C1	69.0 (3)
C11—N2—C14—C13	56.4 (2)		

H atoms were treated as riding, with C—H = 0.95 (6) Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *CrystalClear* (Rigaku/MSC, 2002); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2003); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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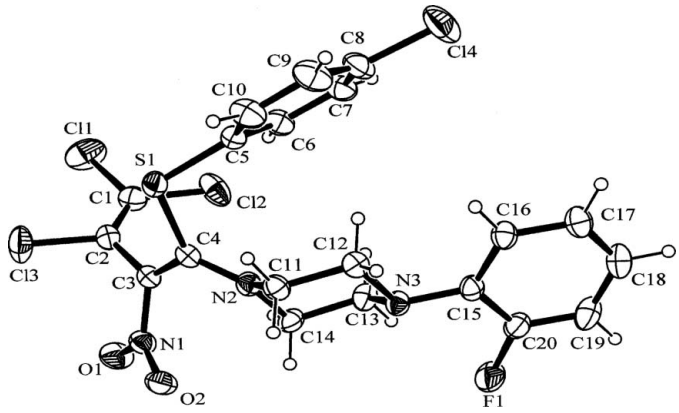

Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

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