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3,4,4-Trichloro-1-[4-(2-fluorophenyl)piperazinyl]-1-(*n*-hexadecylsulfanyl)-2-nitrobuta-1,3-diene

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Key indicators

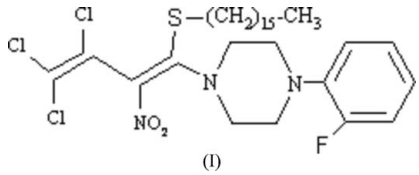
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.057
wR factor = 0.069
Data-to-parameter ratio = 22.7

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

In the title compound, $C_{30}H_{45}Cl_3FN_3O_2S$, the piperazine ring is in a chair conformation. The alkyl substituent has a fully extended conformation.

Comment

Piperazine compounds are important in clinical chemistry (Soladin & Heat, 1996) and in transfer reactions (Zhao & Miller, 1996). It is known that *N*,*S*-disubstituted diene compounds can be prepared from the reactions of some mono(thio)-substituted compounds with amines (primary amine, piperazine, morpholine, piperidine *etc.*) (Ibis *et al.*, 2001). The title compound, (I), was synthesized from 1,3,4,4-tetrachloro-1-(hexadecylsulfanyl)-2-nitrobuta-1,3-diene and 1-(2-fluorophenyl)piperazine (Ibis, Sayil & Ozkok, 2006).



(I)

The structure of (I) (Fig. 1) contains the expected *N*,*S*-disubstituted butadienyl skeleton. The butadiene unit has assumed a configuration close to *cisoid*, but is not completely planar as it would be if the two double bonds were fully conjugated. In the structure of (I), the U_{eq} values of the C atoms of the hexadecyl chain generally increase on going from C15 to C30, reflecting libration of the chain. For the chain C15–C30, the average C—C bond length is 1.514 (4) Å and the average C—C—C angle is 114.2 (2)°. All the torsion angles within the hexadecyl chain deviate from 180° by less than 1.0°. Thus, the C_{16} chain has a near *trans*-coplanar conformation. The observed values are consistent with the corresponding values in a similar compound (Jaeger *et al.*, 1998). The piperazine ring is in a chair conformation. The C—C bond lengths of the butadiene unit are 1.406, 1.452 and 1.334 Å, respectively, for C3—C4, C3—C2 and C2—C1 (Surange *et al.*, 1997; Ibis, Sayil & Deniz, 2006).

Experimental

1,3,4,4-Tetrachloro-1-(hexadecylsulfanyl)-2-nitrobuta-1,3-diene (0.309 g, 0.6 mmol) and 1-(2-fluorophenyl)piperazine (0.112 g, 0.6 mmol) were mixed in dichloromethane (25 ml) at room temperature. The mixture was stirred for 2–3 h. Chloroform was added to the reaction mixture. The organic layer was separated and washed with water (4 × 30 ml) and dried with Na_2SO_4. After the solvent had evaporated, the residue was purified by column chromatography on silica gel. Orange crystals of (I) suitable for X-ray

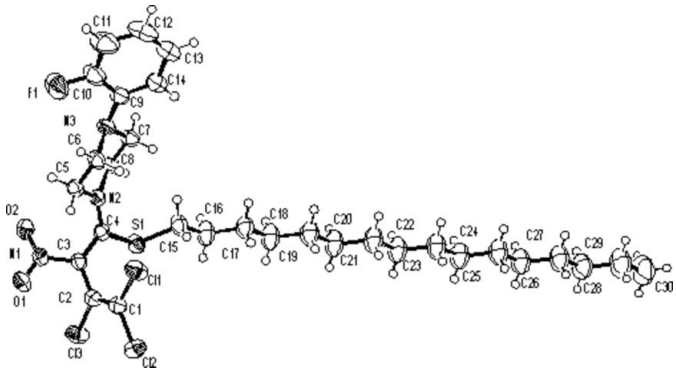


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

diffraction analysis were obtained by slow evaporation of an ethanol at room temperature (yield: 0.14 g, 38%; m.p.362–363 K).

Crystal data

$C_{30}H_{45}Cl_3FN_3O_2S$
$M_r = 637.12$
Triclinic, $P\overline{1}$
$a = 7.0852\ (4)$ Å
$b = 8.3609\ (2)$ Å
$c = 30.031\ (2)$ Å
$\alpha = 83.958\ (7)^\circ$
$\beta = 89.631\ (9)^\circ$
$\gamma = 71.030\ (7)^\circ$
$V = 1672.30\ (17)$ Å^3
$Z = 2$
$D_x = 1.265$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12683 reflections
$\theta = 2.7$–30.0°
$\mu = 0.37$ mm^{-1}
$T = 293.5$ K
Block, orange
$0.40 \times 0.40 \times 0.20$ mm

Data collection

Rigaku R-AXIS diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.867$, $T_{max} = 0.930$
91368 measured reflections
9942 independent reflections
8220 reflections with $F^2 > 2\sigma(F^2)$
$R_{int} = 0.033$
$\theta_{max} = 30.2^\circ$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -42 \rightarrow 42$

Refinement

Refinement on F
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.069$
$S = 1.05$
8220 reflections
361 parameters
All H-atom parameters constrained
Chebychev polynomial *w*ith 3 parameters (Carruthers & Watkin, 1979): 8.9436, 3.9410 and 6.7084
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.85$ e Å^{-3}
$\Delta\rho_{min} = -0.55$ e Å^{-3}

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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