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3,4,4-Trichloro-2-nitro-1-propylsulfanyl-1-(4-thiomorpholinyl)buta-1,3-diene

Cemil Ibis* and N. Gulsah Deniz

Istanbul University, Faculty of Engineering, Department of Chemistry, 34320 Avcilar-Istanbul, Turkey

Correspondence e-mail: ibiscml@istanbul.edu.tr

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.008 Å
R factor = 0.075
wR factor = 0.134
Data-to-parameter ratio = 15.5

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

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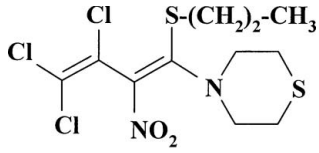
In the title compound, $C_{11}H_{15}Cl_3N_2O_2S_2$, the thiomorpholine ring adopts a chair conformation and the butadiene has a conformation closer to *cisoid* than to *transoid*.

Comment

Thiomorpholine analogues have found applications in medicine and agriculture (Barbacyn *et al.*, 1996). Subsituted thiomorpholino, morpholino and piperidino compounds enhanced the activity against Gram-positive bacteria, but reduced the activity against Gram-negative bacteria (Taguchi *et al.*, 1992). The aim of this study was to determine the conformation of the title compound, (I).



(I)

The structure of (I) contains the expected *N*,*S*-substituted butadienyl skeleton, an alkylsulfanyl chain and a thiomorpholine ring (Fig. 1). The butadiene has a conformation

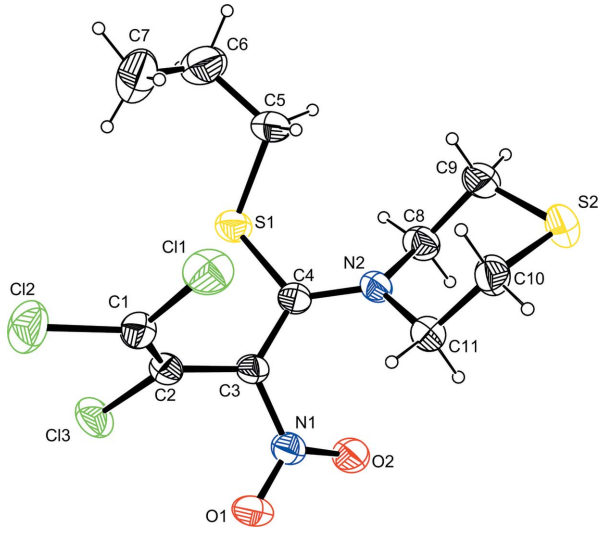


Figure 1
The molecular structure of (I), showing the atom labelling. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented as small spheres of arbitrary radii.

closer to *cisoid* than to *transoid*, the C4—C3—C2—C1 torsion angle being −64.3 (3)°. The C—C bond lengths within the butadiene unit are similar to those in related compounds (Surange *et al.*, 1997; Ibis *et al.*, 2006). The thiomorpholine ring adopts a chair conformation, as shown by the puckering angles of φ = 1(3)° and θ = 10.2 (4)° (Cremer & Pople, 1975).

Experimental

The title compound, (I), was synthesized from 2-nitro-1,3,4,4-tetrachloro-1-(propylsulfanyl)-1,3-butadiene and thiomorpholine (Ibis & Deniz, 2006). Equimolar amounts of 2-nitro-1,3,4,4-tetrachloro-1-(propylsulfanyl)-1,3-butadiene (0.4 g, 1.28 mmol) and thiomorpholine (0.13 g, 1.28 mmol) were stirred in dichloromethane until completion of the reaction. Chloroform (50 ml) was added to the reaction mixture. The organic layer was separated, washed with water (4 × 30 ml) and dried with $CaCl_2$ or $MgSO_4$. After the solvent had evaporated, the residue was purified by column chromatography on silica gel. Yellow crystals of (I) suitable for X-ray diffraction analysis were obtained by slow evaporation of an ethanol solution at room temperature (yield: 0.26 g, 54%; m.p. 424–426 K).

Crystal data

$C_{11}H_{15}Cl_3N_2O_2S_2$
M_r = 377.73
Monoclinic, $P2_1/n$
a = 11.3127 (5) Å
b = 9.1100 (3) Å
c = 16.3114 (8) Å
β = 102.803 (2)°
V = 1639.24 (12) Å^3
Z = 4
D_x = 1.530 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.81 mm^{-1}
T = 293.5 K
Platelet, yellow
0.40 × 0.20 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID S diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.820, T_{max} = 0.852
63776 measured reflections
2824 independent reflections
2824 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 25.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.075
$wR(F^2)$ = 0.134
S = 1.48
2824 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0165P)^2 + 2.4574P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

All H atoms were positioned geometrically and treated as riding, with C—H = 0.93 (CH_3) or 0.97 Å (CH_2), with $U_{iso}(H) = 1.2U_{eq}(CH_2)$ and $1.5U_{eq}(CH_3)$.

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: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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