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

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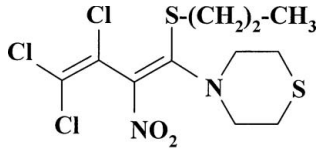
In the title compound, $C_{11}H_{15}Cl_3N_2O_2S_2$, the thiomorpholine ring adopts a chair conformation and the butadiene has a conformation closer to *cisoid* than to *transoid*.

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

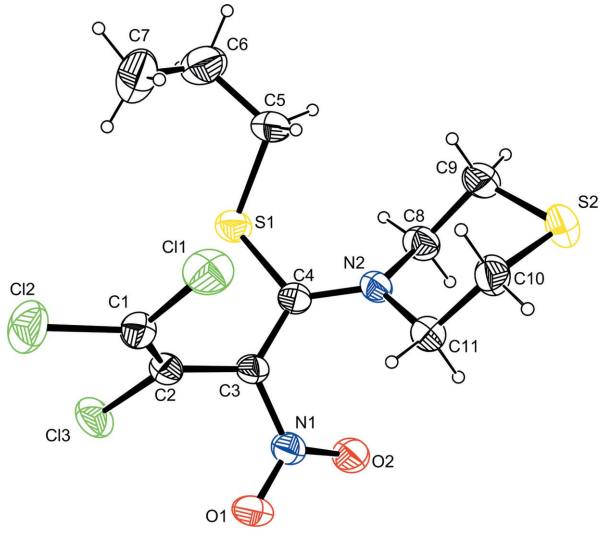


Figure 1
The molecular structure of (I), showing the atom labelling. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented as small spheres of arbitrary radii.

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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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$M_r = 377.73$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 11.3127$ (5) Å
$b = 9.1100$ (3) Å
$c = 16.3114$ (8) Å
$\beta = 102.803$ (2)°
$V = 1639.24$ (12) Å^3
$Z = 4$
$F(000) = 776.00$
$D_x = 1.530$ Mg m^{-3}
Melting point: 425 K
Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å
Cell parameters from 9300 reflections
$\theta = 2.8$–$25.3°$
$\mu = 0.81$ mm^{-1}
$T = 294$ K
Platelet, yellow
0.40 × 0.20 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID S diffractometer
Detector resolution: 10.00 pixels mm^{-1}
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 $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 25.2°$, $\theta_{min} = 2.9°$
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.075$
$wR(F^2) = 0.134$
$S = 1.48$
2824 reflections
182 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
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}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl3	0.13819 (11)	0.02140 (17)	0.04376 (9)	0.0742 (4)
Cl1	0.52310 (12)	−0.02457 (18)	0.11238 (10)	0.0826 (5)
Cl2	0.32805 (17)	−0.23042 (16)	0.06265 (11)	0.0917 (5)
S1	0.34076 (11)	0.17623 (16)	0.25363 (8)	0.0609 (4)
S2	0.69722 (14)	0.57653 (18)	0.23389 (11)	0.0826 (5)
O2	0.2925 (3)	0.4442 (4)	0.0343 (2)	0.0685 (10)
O1	0.2546 (3)	0.2564 (4)	−0.0487 (2)	0.0716 (10)
N2	0.4659 (3)	0.3734 (4)	0.1834 (2)	0.0479 (9)
N1	0.2916 (3)	0.3094 (4)	0.0222 (2)	0.0549 (10)
C3	0.3273 (4)	0.2124 (5)	0.0907 (3)	0.0452 (10)
C11	0.5327 (4)	0.4140 (6)	0.1189 (3)	0.0561 (12)
H11A	0.4998	0.5046	0.0919	0.067*
H11B	0.5224	0.3380	0.0763	0.067*
C4	0.3840 (4)	0.2649 (5)	0.1701 (3)	0.0459 (10)
C8	0.4765 (5)	0.4775 (6)	0.2535 (3)	0.0618 (13)
H8A	0.4299	0.4410	0.2924	0.074*
H8B	0.4420	0.5709	0.2318	0.074*
C2	0.2928 (4)	0.0599 (5)	0.0731 (3)	0.0497 (11)
C9	0.6068 (5)	0.5014 (6)	0.3005 (3)	0.0742 (16)
H9A	0.6412	0.4084	0.3231	0.089*
H9B	0.6080	0.5675	0.3473	0.089*
C1	0.3701 (5)	−0.0499 (5)	0.0803 (3)	0.0577 (12)
C10	0.6661 (4)	0.4340 (6)	0.1570 (3)	0.0632 (13)
H10A	0.7078	0.4560	0.1125	0.076*
H10B	0.6986	0.3424	0.1829	0.076*
C5	0.4808 (5)	0.1548 (7)	0.3338 (3)	0.0745 (16)
H5A	0.5492	0.1492	0.3070	0.089*
H5B	0.4923	0.2396	0.3707	0.089*
C6	0.4764 (7)	0.0199 (9)	0.3840 (4)	0.103 (2)
H6A	0.5465	0.0196	0.4307	0.124*
H6B	0.4048	0.0248	0.4075	0.124*
C7	0.4738 (8)	−0.1176 (8)	0.3396 (6)	0.127 (3)
H7A	0.3987	−0.1259	0.2987	0.191*
H7B	0.4809	−0.1974	0.3787	0.191*
H7C	0.5402	−0.1207	0.3117	0.191*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl3	0.0490 (7)	0.0909 (10)	0.0767 (9)	−0.0181 (7)	0.0010 (6)	−0.0063 (8)

Cl1	0.0565 (8)	0.0912 (11)	0.0983 (11)	0.0222 (7)	0.0131 (7)	0.0130 (9)
Cl2	0.1273 (14)	0.0484 (8)	0.1033 (12)	−0.0018 (8)	0.0338 (10)	−0.0062 (8)
S1	0.0537 (7)	0.0801 (9)	0.0477 (6)	−0.0081 (6)	0.0086 (5)	0.0066 (6)
S2	0.0738 (9)	0.0733 (10)	0.0913 (11)	−0.0252 (8)	−0.0021 (8)	−0.0032 (8)
O2	0.078 (2)	0.048 (2)	0.069 (2)	0.0069 (17)	−0.0069 (18)	0.0042 (17)
O1	0.086 (3)	0.073 (2)	0.0442 (19)	−0.002 (2)	−0.0092 (17)	0.0018 (18)
N2	0.049 (2)	0.051 (2)	0.0417 (19)	−0.0047 (17)	0.0055 (16)	−0.0045 (17)
N1	0.056 (2)	0.052 (2)	0.051 (2)	0.0007 (19)	−0.0002 (18)	−0.0002 (19)
C3	0.041 (2)	0.047 (2)	0.043 (2)	0.0032 (19)	0.0012 (18)	0.003 (2)
C11	0.058 (3)	0.056 (3)	0.052 (3)	−0.005 (2)	0.008 (2)	−0.002 (2)
C4	0.040 (2)	0.050 (3)	0.047 (2)	0.005 (2)	0.0079 (18)	0.001 (2)
C8	0.067 (3)	0.061 (3)	0.054 (3)	0.001 (3)	0.006 (2)	−0.008 (2)
C2	0.042 (2)	0.055 (3)	0.048 (2)	−0.002 (2)	0.0030 (19)	0.002 (2)
C9	0.089 (4)	0.068 (4)	0.056 (3)	−0.011 (3)	−0.005 (3)	−0.007 (3)
C1	0.066 (3)	0.051 (3)	0.055 (3)	−0.002 (2)	0.011 (2)	0.000 (2)
C10	0.052 (3)	0.060 (3)	0.076 (3)	−0.006 (2)	0.011 (2)	0.003 (3)
C5	0.073 (4)	0.090 (4)	0.052 (3)	−0.010 (3)	−0.006 (3)	0.014 (3)
C6	0.095 (5)	0.118 (6)	0.089 (5)	−0.010 (4)	0.007 (4)	0.041 (5)
C7	0.131 (7)	0.078 (5)	0.164 (8)	−0.018 (5)	0.013 (6)	0.026 (6)

Geometric parameters (Å, º)

Cl3—C2	1.744 (4)	C8—C9	1.518 (7)
Cl1—C1	1.710 (5)	C8—H8A	0.9700
Cl2—C1	1.719 (5)	C8—H8B	0.9700
S1—C4	1.744 (4)	C2—C1	1.316 (6)
S1—C5	1.828 (5)	C9—H9A	0.9700
S2—C10	1.785 (5)	C9—H9B	0.9700
S2—C9	1.786 (6)	C10—H10A	0.9700
O2—N1	1.243 (5)	C10—H10B	0.9700
O1—N1	1.238 (5)	C5—C6	1.484 (8)
N2—C4	1.340 (5)	C5—H5A	0.9700
N2—C8	1.470 (6)	C5—H5B	0.9700
N2—C11	1.472 (6)	C6—C7	1.444 (10)
N1—C3	1.411 (5)	C6—H6A	0.9700
C3—C4	1.397 (6)	C6—H6B	0.9700
C3—C2	1.454 (6)	C7—H7A	0.9600
C11—C10	1.511 (6)	C7—H7B	0.9600
C11—H11A	0.9700	C7—H7C	0.9600
C11—H11B	0.9700		
C4—S1—C5	105.1 (2)	S2—C9—H9A	109.3
C10—S2—C9	95.6 (3)	C8—C9—H9B	109.3
C4—N2—C8	122.4 (4)	S2—C9—H9B	109.3
C4—N2—C11	121.1 (4)	H9A—C9—H9B	107.9
C8—N2—C11	115.3 (4)	C2—C1—Cl1	122.2 (4)
O1—N1—O2	121.5 (4)	C2—C1—Cl2	124.0 (4)
O1—N1—C3	118.3 (4)	Cl1—C1—Cl2	113.8 (3)

O2—N1—C3	120.1 (4)	C11—C10—S2	113.6 (4)
C4—C3—N1	120.8 (4)	C11—C10—H10A	108.8
C4—C3—C2	123.9 (4)	S2—C10—H10A	108.8
N1—C3—C2	115.2 (4)	C11—C10—H10B	108.8
N2—C11—C10	111.1 (4)	S2—C10—H10B	108.8
N2—C11—H11A	109.4	H10A—C10—H10B	107.7
C10—C11—H11A	109.4	C6—C5—S1	110.7 (4)
N2—C11—H11B	109.4	C6—C5—H5A	109.5
C10—C11—H11B	109.4	S1—C5—H5A	109.5
H11A—C11—H11B	108.0	C6—C5—H5B	109.5
N2—C4—C3	124.1 (4)	S1—C5—H5B	109.5
N2—C4—S1	121.3 (3)	H5A—C5—H5B	108.1
C3—C4—S1	114.6 (3)	C7—C6—C5	116.1 (6)
N2—C8—C9	112.9 (4)	C7—C6—H6A	108.3
N2—C8—H8A	109.0	C5—C6—H6A	108.3
C9—C8—H8A	109.0	C7—C6—H6B	108.3
N2—C8—H8B	109.0	C5—C6—H6B	108.3
C9—C8—H8B	109.0	H6A—C6—H6B	107.4
H8A—C8—H8B	107.8	C6—C7—H7A	109.5
C1—C2—C3	124.4 (4)	C6—C7—H7B	109.5
C1—C2—Cl3	118.5 (4)	H7A—C7—H7B	109.5
C3—C2—Cl3	117.1 (3)	C6—C7—H7C	109.5
C8—C9—S2	111.7 (4)	H7A—C7—H7C	109.5
C8—C9—H9A	109.3	H7B—C7—H7C	109.5