

Table 2. Selected geometric parameters (Å, °)

N(1)—C(1)	1.342 (5)	N(1)—C(2)	1.332 (5)
N(2)—C(3)	1.433 (4)	N(2)—C(6)	1.349 (5)
N(3)—C(6)	1.320 (5)	N(3)—C(7)	1.452 (6)
N(4)—C(6)	1.320 (5)	N(4)—C(14)	1.306 (6)
N(5)—C(14)	1.149 (6)	N(6)—C(1)	1.364 (5)
N(7)—C(15)	1.104 (6)	C(1)—C(5)	1.397 (6)
C(2)—C(3)	1.362 (5)	C(3)—C(4)	1.398 (5)
C(4)—C(5)	1.361 (5)	C(7)—C(8)	1.547 (7)
C(7)—C(12)	1.54 (1)	C(8)—C(9)	1.502 (7)
C(9)—C(10)	1.48 (1)	C(9)—C(13)	1.47 (1)
C(10)—C(11)	1.50 (2)	C(11)—C(12)	1.49 (1)
C(12)—C(13)	1.55 (1)	C(15)—C(16)	1.456 (8)
C(1)—N(1)—C(2)	117.0 (3)	C(3)—N(2)—C(6)	124.2 (3)
C(6)—N(3)—C(7)	122.2 (3)	C(6)—N(4)—C(14)	120.0 (3)
N(1)—C(1)—N(6)	117.3 (3)	N(1)—C(1)—C(5)	122.3 (3)
N(6)—C(1)—C(5)	120.3 (3)	N(1)—C(2)—C(3)	124.8 (3)
N(2)—C(3)—C(2)	120.9 (3)	N(2)—C(3)—C(4)	121.1 (3)
C(2)—C(3)—C(4)	117.8 (3)	C(3)—C(4)—C(5)	119.0 (3)
C(1)—C(5)—C(4)	119.1 (3)	N(2)—C(6)—N(3)	119.6 (3)
N(2)—C(6)—N(4)	122.3 (4)	N(3)—C(6)—N(4)	118.1 (3)
N(3)—C(7)—C(8)	112.8 (4)	N(3)—C(7)—C(12)	115.5 (5)
C(8)—C(7)—C(12)	101.2 (4)	C(7)—C(8)—C(9)	103.3 (5)
C(8)—C(9)—C(10)	106.0 (5)	C(8)—C(9)—C(13)	104.1 (5)
C(10)—C(9)—C(13)	103.3 (8)	C(9)—C(10)—C(11)	103.5 (8)
C(10)—C(11)—C(12)	103.5 (8)	C(7)—C(12)—C(11)	108.9 (5)
C(7)—C(12)—C(13)	99.6 (7)	C(11)—C(12)—C(13)	101.6 (8)
C(9)—C(13)—C(12)	93.7 (5)	N(4)—C(14)—N(5)	173.2 (4)
N(7)—C(15)—C(16)	175.7 (8)		
N(1)—C(1)—C(5)—C(4)	0.6 (6)		
N(1)—C(2)—C(3)—C(4)	0.5 (6)		
N(2)—C(6)—N(3)—C(7)	177.6 (4)		
N(3)—C(6)—N(2)—C(3)	2.6 (5)		
N(3)—C(7)—C(8)—C(9)	-130.7 (5)		
N(3)—C(7)—C(12)—C(13)	162.3 (5)		
N(4)—C(6)—N(3)—C(7)	-2.2 (6)		
N(6)—C(1)—N(1)—C(2)	177.3 (4)		
C(1)—N(1)—C(2)—C(3)	1.2 (6)		
C(2)—N(1)—C(1)—C(5)	-1.8 (6)		
C(2)—C(3)—C(4)—C(5)	-1.6 (6)		
C(6)—N(3)—C(7)—C(8)	-162.9 (4)		
C(7)—C(8)—C(9)—C(10)	77.0 (7)		
C(7)—C(12)—C(11)—C(10)	72.3 (8)		
C(8)—C(7)—C(12)—C(11)	-65.8 (8)		
C(8)—C(9)—C(10)—C(11)	-71.7 (8)		
C(9)—C(8)—C(7)—C(12)	-6.7 (6)		
C(9)—C(13)—C(12)—C(11)	53.2 (8)		
C(10)—C(11)—C(12)—C(13)	-32.2 (8)		
N(1)—C(2)—C(3)—N(2)	-174.0 (4)		
N(2)—C(3)—C(4)—C(5)	172.8 (4)		
N(2)—C(6)—N(4)—C(14)	-2.8 (6)		
N(3)—C(6)—N(4)—C(14)	177.0 (4)		
N(3)—C(7)—C(12)—C(11)	56.4 (8)		
N(4)—C(6)—N(2)—C(3)	-177.6 (3)		
N(5)—C(14)—N(4)—C(6)	173 (3)		
N(6)—C(1)—C(5)—C(4)	-178.4 (4)		
C(1)—C(5)—C(4)—C(3)	1.1 (6)		
C(2)—C(3)—N(2)—C(6)	-90.9 (4)		
C(4)—C(3)—N(2)—C(6)	94.8 (5)		
C(6)—N(3)—C(7)—C(12)	81.4 (6)		
C(7)—C(8)—C(9)—C(13)	-31.6 (7)		
C(7)—C(12)—C(13)—C(9)	-58.5 (7)		
C(8)—C(7)—C(12)—C(13)	40.1 (7)		
C(8)—C(9)—C(13)—C(12)	55.4 (8)		
C(9)—C(10)—C(11)—C(12)	-1.7 (9)		
C(10)—C(9)—C(13)—C(12)	-55.1 (8)		
C(11)—C(10)—C(9)—C(13)	37.4 (7)		

The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985) and expanded by Fourier techniques using *DIRDIF92* (Beurskens *et al.*, 1992). The structure was refined by a full-matrix least-squares procedure. All calculations were performed using *TEXSAN* (Molecular Structure Corporation, 1992).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: OH1073). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(5-chloro-2-nitrophenyl) Disulfide

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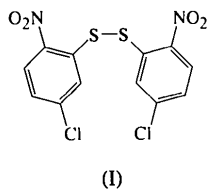
Abstract

The title molecule, $C_{12}H_6Cl_2N_2O_4S_2$, lies on a twofold axis in the crystal. The C—S—S—C group has a skewed non-planar conformation with a torsion-angle magnitude of $86.7(1)^\circ$. The S—S—C bond angle is $104.11(5)^\circ$, and the S—S, S—C, C—N and C—Cl bond lengths are 2.0432(5), 1.789(1), 1.462(2) and 1.726(1) Å, respectively. The nitro group is rotated $15.2(2)^\circ$ out of the plane of the phenyl ring.

Comment

The title compound, (I), consists of two identical monomeric units linked by a disulfide bridge lying on a twofold axis. The torsion angle about the S—S bond is $86.7(1)^\circ$.

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The molecule adopts a skewed non-planar conformation similar to that found in H₂O₂ (Abrahams, Collin & Lipscomb, 1951), H₂S₂ (Wilson & Badger, 1949), (CH₃)₂S₂ (Stevenson & Beach, 1938), (C₆H₅)₂S₂ (Lee & Bryant, 1969*a*) and (C₆H₅CH₂)₂S₂ (Lee & Bryant, 1969*b*). The S—S bond length [2.0432 (5) Å] is slightly longer than the mean value of 2.029 Å for 46 disulfides with C—S—S—C torsion angles in the range 75–105° (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987), but shorter than the mean value of 2.068 Å for 28 disulfides with C—S—S—C angles in the range 0–20°. The C—S distance of 1.789 (1) Å agrees with that of diphenyl disulfide (Lee & Bryant, 1969*a*) and is slightly shorter than the average value for 11 molecules containing a single S—S bond (1.812 Å) obtained by Cox & Jeffrey (1951) and those of dibenzyl disulfide (1.85 and 1.84 Å; Lee & Bryant 1969*b*). The crystal packing is illustrated in Fig. 2. The aromatic rings stack in pairs related by inversion centers [at ($\frac{1}{4}, \frac{1}{4}, \frac{1}{2}$) and equivalent sites], with distances of 3.647 Å between the centroids of the rings.

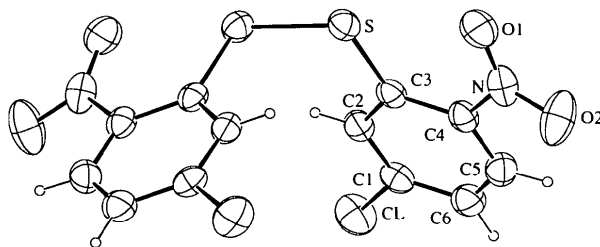


Fig. 1. The molecular structure of the title compound showing 40% probability ellipsoids. H atoms are illustrated by circles of arbitrary radii.

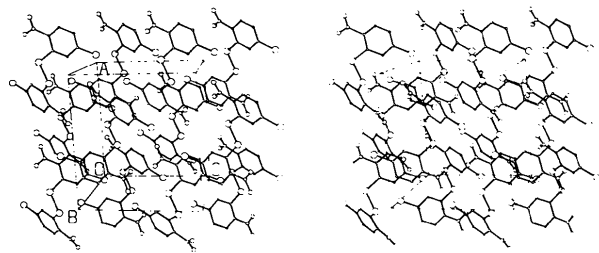


Fig. 2. Stereoview of the unit cell slightly oblique to the **b** direction.

Experimental

Bis(5-chloro-2-nitrophenyl) disulfide, (I), was formed as a by-product when 5-chloro-2-nitrobenzenethiol (Bourdais, 1966) was allowed to react with chloroisonicotinic acid (Haydar, Garcia & Krapcho, 1995).

Crystal data

C₁₂H₆Cl₂N₂O₄S₂

M_r = 377.23

Monoclinic

C2/c

a = 11.5304 (6) Å

b = 12.2492 (5) Å

c = 10.2645 (6) Å

β = 91.89 (1)°

V = 1448.9 (2) Å³

Z = 4

D_x = 1.728 Mg m⁻³

Mo Kα radiation

λ = 0.71073 Å

Cell parameters from 25

reflections

θ = 10–13°

μ = 0.741 mm⁻¹

T = 296 K

Fragment

0.50 × 0.35 × 0.32 mm

Golden yellow

Data collection

Enraf–Nonius CAD-4
diffractometer

θ/2θ scans

Absorption correction:

ψ scans (North, Phillips
& Mathews, 1968)

T_{min} = 0.9497, *T_{max}* =
0.9991

2943 measured reflections

2728 independent reflections

2056 observed reflections

[*I* > 3σ(*I*)]

R_{int} = 0.01

θ_{max} = 32.96°

h = 0 → 17

k = 0 → 18

l = -15 → 15

3 standard reflections

frequency: 120 min

intensity decay: 0.01%

Refinement

Refinement on *F*

R = 0.033

wR = 0.044

S = 2.173

2056 reflections

113 parameters

H atoms refined isotropically

w = 4*F_o*²/[σ²(*F_o*²)
+ 0.0004*F_o*⁴]

(Δ/σ)_{max} = 0.019

Δρ_{max} = 0.281 e Å⁻³

Δρ_{min} = -0.261 e Å⁻³

Extinction correction:

isotropic (Zachariasen,
1963)

Extinction coefficient:

0.13 (2) × 10⁻⁵

Atomic scattering factors

from *International Tables*
for X-ray Crystallography
(1974, Vol. IV)

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

*U*_{iso} for H atoms; *U*_{eq} = (1/3)Σ_{*i*}Σ_{*j*}*U*_{*ij*}*a*_{*i*}^{*}*a*_{*j*}^{*} for others.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} / <i>U</i> _{iso}
Cl	0.10645 (4)	0.45630 (4)	0.62491 (4)	0.0634 (1)
S	0.08186 (3)	0.17375 (3)	0.21485 (4)	0.04381 (8)
O1	0.2881 (1)	0.1829 (1)	0.1155 (1)	0.0588 (3)
O2	0.4083 (1)	0.3175 (1)	0.1220 (1)	0.0797 (4)
N	0.3252 (1)	0.2677 (1)	0.1635 (1)	0.0494 (3)
C1	0.1684 (1)	0.4024 (1)	0.4882 (1)	0.0427 (3)
C2	0.1082 (1)	0.3240 (1)	0.4169 (1)	0.0401 (3)
C3	0.1564 (1)	0.2767 (1)	0.3084 (1)	0.0359 (3)
C4	0.2667 (1)	0.3128 (1)	0.2756 (1)	0.0389 (3)
C5	0.3259 (1)	0.3925 (1)	0.3468 (2)	0.0474 (4)
C6	0.2775 (1)	0.4375 (1)	0.4544 (2)	0.0483 (4)
H2	0.040 (1)	0.303 (1)	0.443 (2)	0.055 (5)
H5	0.399 (1)	0.414 (2)	0.320 (2)	0.056 (5)
H6	0.317 (1)	0.489 (1)	0.510 (2)	0.049 (4)

Table 2. Selected geometric parameters (Å, °)

Cl—C1	1.726 (1)	C2—C3	1.388 (2)
S—S ¹	2.0432 (5)	C3—C4	1.398 (2)
S—C3	1.789 (1)	C4—C5	1.386 (2)
O1—N	1.221 (2)	C5—C6	1.370 (2)
O2—N	1.224 (2)	C2—H2	0.88 (2)
N—C4	1.462 (2)	C5—H5	0.93 (2)
C1—C2	1.381 (2)	C6—H6	0.96 (2)
C1—C6	1.384 (2)		
S—S ¹ —C3	104.11 (5)	S—C3—C2	121.8 (1)
O1—N—O2	123.5 (1)	S—C3—C4	121.2 (1)
O1—N—C4	118.3 (1)	C2—C3—C4	117.0 (1)
O2—N—C4	118.2 (1)	N—C4—C3	121.2 (1)
Cl—C1—C2	118.8 (1)	N—C4—C5	116.7 (1)
Cl—C1—C6	119.3 (1)	C3—C4—C5	122.1 (1)
C2—C1—C6	121.9 (1)	C4—C5—C6	120.1 (1)
C1—C2—C3	120.5 (1)	C1—C6—C5	118.4 (1)

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

Data collection: CAD-4 diffractometer software (Enraf-Nonius, 1977). Cell refinement: CAD-4 diffractometer software. Data reduction: *MolEN PROCESS* (Fair, 1990). Program(s) used to solve structure: *MULTAN80* (Main *et al.*, 1980). Program(s) used to refine structure: *MolEN LSFM*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *MolEN CIF IN*.

The purchase of the diffractometer was made possible by a National Science Foundation Chemical Instrumentation Grant, which we gratefully acknowledge.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1145). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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endo-5-Methyl-*exo*-6-phenylseleno-*endo*-tricyclo[5.2.1.0^{2,6}]dec-8-en-3-one

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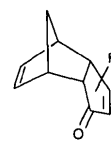
(Received 26 August 1994; accepted 11 May 1995)

Abstract

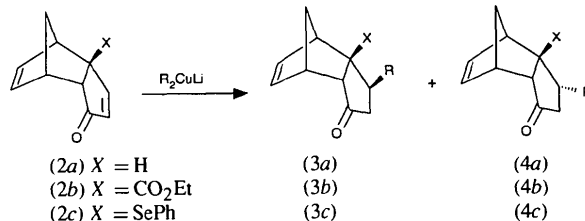
Structural analysis of the title compound, C₁₇H₁₈OSe, confirms the *endo* configuration of the C5-methyl group.

Comment

Tricyclo[5.2.1.0^{2,6}]decadienones, (1), have great potential as synthetic equivalents of cyclopentadienone and serve as building blocks in cyclopentanoid natural-product synthesis (Klunder, Bos & Zwanenburg, 1981; Klunder, Zwanenburg & Liu, 1991; Takano, Moriya & Ogasawara, 1992; Liu & Chu, 1993; Zhu, Klunder & Zwanenburg, 1994a). The *endo*-tricyclodecadienone system, which is racemic and optically active, is also readily accessible *via* the ester (2b).



(1)



Recently, we studied the nucleophilic addition of a variety of alkyl lithium cuprates to the enone double bond in the parent compound (2a), and the ester (2b). Whereas addition to (2a) proceeds with exclusively *exo* addition to give (3a), the presence of the *exo* ester