582 reflections
100 parameters
H-atom parameters not refined

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

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Compound (1)				
S1	0.6401 (1)	0.0984 (1)	0.40165 (7)	3.93 (2)
S2	0.7957 (1)	0.3555 (1)	0.52468 (7)	4.13 (2)
N3	0.3891 (4)	0.3054 (3)	0.4347 (2)	3.35 (6)
C5	0.3599 (5)	0.0801 (4)	0.3394 (3)	3.41 (7)
C4	0.2326 (5)	0.2139 (4)	0.3724 (2)	3.58 (7)
C2	0.5947 (5)	0.2645 (4)	0.4559 (2)	3.00 (7)
C6	0.3441 (6)	0.0887 (4)	0.2240 (3)	3.92 (8)
C7	0.2981 (6)	0.2466 (4)	0.2001 (3)	3.96 (8)
C8	0.1431 (6)	0.2886 (4)	0.2738 (3)	4.51 (8)
Compound (2)				
C2	0.4821 (6)	0.0849 (3)	0.2986 (2)	2.97 (6)
C4	0.1835 (5)	0.2366 (3)	0.3401 (2)	2.77 (6)
C5	0.1584 (6)	0.1352 (2)	0.4048 (2)	3.00 (6)
C3	0.3069 (7)	0.2271 (4)	0.1965 (2)	4.35 (8)
C6	0.3253 (6)	0.3562 (3)	0.3665 (2)	3.34 (7)
C8	0.2567 (7)	0.3065 (4)	0.5091 (2)	4.22 (8)
N3	0.3166 (5)	0.1723 (3)	0.2759 (1)	2.87 (5)
C7	0.2277 (7)	0.4076 (4)	0.4446 (2)	4.09 (8)
S1	0.4534 (2)	0.04754 (8)	0.39948 (5)	3.51 (2)
S2	0.7001 (2)	0.0169 (1)	0.24372 (6)	4.27 (2)
C9	0.1039 (6)	0.1885 (4)	0.4869 (2)	3.92 (7)
Compound (3)				
S2	0.7680 (3)	0.0806 (2)	0.3857 (3)	4.21 (5)
S1	0.8758 (3)	0.3110 (2)	0.4174 (3)	4.83 (6)
N3	1.0598 (7)	0.1529 (5)	0.4653 (7)	3.3 (2)
C2	0.9108 (8)	0.1717 (7)	0.4249 (8)	2.9 (2)
C7	1.283 (1)	0.4596 (7)	0.330 (1)	4.8 (2)
C4	1.1669 (8)	0.2427 (7)	0.5091 (8)	3.0 (2)
C10	1.3230 (9)	0.2181 (6)	0.455 (1)	3.8 (2)
C9	1.4431 (9)	0.3109 (7)	0.480 (1)	4.3 (2)
C8	1.430 (1)	0.3907 (7)	0.350 (1)	4.3 (2)
C6	1.127 (1)	0.3973 (7)	0.302 (1)	4.3 (2)
C5	1.0833 (9)	0.3462 (6)	0.4460 (9)	3.4 (2)

Table 2. *Selected geometric parameters* (Å, °)

	(1)	(2)	(3)
S1—C2	1.732 (4)	1.751 (3)	1.737 (8)
S1—C5	1.819 (3)	1.830 (3)	1.825 (8)
S2—C2	1.666 (3)	1.665 (3)	1.664 (8)
N3—C2	1.313 (3)	1.321 (4)	1.306 (9)
N3—C4	1.456 (4)	1.471 (4)	1.46 (1)
C4—C5	1.554 (5)	1.525 (4)	1.53 (1)
C2—S1—C5	94.5 (1)	91.9 (1)	93.8 (4)
S1—C2—S2	121.8 (2)	121.5 (2)	122.3 (4)
S2—C2—N3	126.7 (3)	127.6 (2)	127.6 (6)
C2—N3—C4	120.2 (3)	116.0 (2)	120.2 (6)
N3—C4—C5	107.6 (2)	105.1 (2)	106.5 (6)
C4—C5—S1	106.2 (2)	102.7 (2)	105.0 (5)

Table 3. *Distances* (Å) *to the* S1—C2—S2—N3 *fragment and values of the torsion angle* τ (°)

	(1)	(2)	(3)
C4	0.010 (3)	−0.290 (3)	−0.121 (7)
C5	0.077 (3)	0.315 (3)	0.243 (8)
τ	3.4 (3)	36.6 (2)	21.3 (7)

H atoms were introduced at idealized positions in the calculations before the last refinement cycle, but were not refined.

Program used for data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Program used throughout the analysis: *SDP-Plus* (Frenz, 1985). Program used to solve the structures: *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson (1982) (completed by Fourier synthesis). Molecular graphics: *ORTEP*II (Johnson, 1976).

For all three structures, lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: NA1157). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3,7-Di-*tert*-butyl-1,5,2,4,6,8-dithiatetrazocine

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Abstract

The eight-membered ring in the title compound, $C_{10}H_{18}N_4S_2$, lies about an inversion centre and is essentially planar with normal bond lengths and angles [mean C—S 1.569 (2) and mean C—N 1.326 (3) Å].

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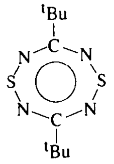
Comment

We are presently engaged in a study of the chemical and electrochemical properties of unsaturated inorganic ring systems. We have reported the electrochemical properties of the title compound, (I) (Boeré *et al.*, 1993), and now report the results of a single-crystal X-ray diffraction study of its structure.



(I)

An *ORTEP*II (Johnson, 1976) illustration of the molecule is presented in Fig. 1, which also gives the atomic numbering scheme. The eight-membered $C_2N_4S_2$ ring is found to be planar [maximum deviation 0.002 (1) Å for N2], as suspected from the similarity of its redox potentials to those of planar aryl derivatives. The bent 3,5-bis(dimethylamino) derivative (Ernst *et al.*, 1981) has very different redox potentials.

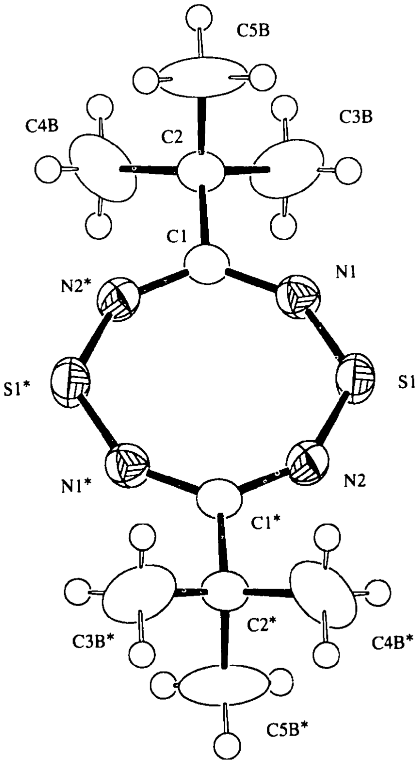


Fig. 1. A view of the title compound with the atom-numbering scheme. Part of the disordered *tert*-butyl group with lower occupancy factor has been ignored. The non-H atoms are plotted as displacement ellipsoids at the 50% probability level.

The structure of the C—N—S ring is essentially similar to those found in other planar dithiatetrazocine derivatives, *i.e.* 4-$CF_3C_6H_4$- (Boeré *et al.*, 1993), C_6H_5- and 4-$CH_3OC_6H_4$- (Ernst *et al.*, 1981) derivatives. The bond lengths [mean S—N 1.569 (2) and mean N—C 1.326 (3) Å] and bond angles [N—S—N 127.20 (12), C—N—S 142.1 (2) and N—C—N 128.6 (2)°] within the heterocyclic ring in the title compound are not significantly different from those found for other structures having aryl rather than alkyl substituents.

It is remarkable that all four structures mentioned above possess $\bar{1}$ site symmetry, even though they crystallize in different space groups.

Experimental

The title compound was prepared according to the method of Gleiter, Bartetzko & Cremer (1984). Crystals were obtained as yellow blocks by sublimation under static vacuum onto a large water-cooled cold finger.

Crystal data

$C_{10}H_{18}N_4S_2$
$M_r = 258.40$
Trigonal
$R\bar{3}$
$a = 19.244$ (7) Å
$c = 9.651$ (2) Å
$V = 3095.2$ (17) Å^3
$Z = 9$
$D_x = 1.248$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 18 reflections
$\theta = 15$–$20°$
$\mu = 0.369$ mm^{-1}
$T = 293$ (1) K
Block
0.52 × 0.40 × 0.35 mm
Yellow

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.853$, $T_{\max} = 1.000$
1360 measured reflections
1220 independent reflections

856 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0176$
$\theta_{\max} = 25.0°$
$h = -19 \rightarrow 19$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 11$
3 standard reflections monitored every 200 reflections intensity decay: 1.55%

Refinement

Refinement on F^2
$R(F) = 0.0384$
$wR(F^2) = 0.1141$
$S = 0.984$
1220 reflections
101 parameters
H atoms refined as riding (C—H 0.96 Å)

$w = 1/[\sigma^2(F_o^2) + (0.1000P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.013$
$\Delta\rho_{\max} = 0.283$ e Å^{-3}
$\Delta\rho_{\min} = -0.182$ e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
S1	0.78680 (4)	0.06464 (4)	0.25216 (8)	0.0412 (3)
N1	0.7272 (1)	0.0909 (1)	0.1951 (2)	0.0360 (6)
N2	0.8809 (1)	0.1136 (1)	0.2459 (2)	0.0366 (6)
C1	0.7249 (2)	0.1504 (2)	0.1306 (2)	0.0315 (6)
C2	0.6410 (2)	0.1377 (2)	0.0996 (3)	0.0380 (7)
C3*A*†	0.5741 (7)	0.0533 (6)	0.1503 (16)	0.056 (3)
C4*A*†	0.6290 (5)	0.1941 (6)	0.2074 (13)	0.066 (3)
C5*A*†	0.6311 (7)	0.1570 (14)	−0.0377 (11)	0.114 (7)
C3*B*‡	0.5819 (6)	0.0856 (8)	0.2038 (10)	0.080 (3)
C4*B*‡	0.6415 (4)	0.2160 (4)	0.0880 (14)	0.083 (4)
C5*B*‡	0.6195 (5)	0.0979 (7)	−0.0472 (9)	0.081 (3)

† Occupancy = 0.42 (1).
‡ Occupancy = 0.58 (1).

Table 2. *Selected geometric parameters* (Å, °)

S1—N1	1.567 (2)	N2—C1^i	1.328 (3)
S1—N2	1.570 (2)	C1—C2	1.537 (3)
N1—C1	1.323 (3)		
N1—S1—N2	127.20 (12)	N1^i—C1—N2	128.6 (2)
C1—N1—S1	142.2 (2)	N1—C1—C2	116.1 (2)
C1^i—N2—S1	142.0 (2)	N2^i—C1—C2	115.3 (2)

Symmetry code: (i) $\frac{5}{3} - x, \frac{1}{3} - y, \frac{1}{3} - z$.

The space group was determined from the systematic *hkl* absences (when $-h + k + l \neq 3n$), a statistical analysis of intensity distribution and the successful solution and refinement of the structure. Methyl C atoms of the *tert*-butyl group were disordered over two sites with unequal occupancy factors [0.42 (1)/0.58(1)].

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *PATTY* in *DIRDIF* (Beurskens *et al.*, 1992). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1068). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Dimer of 4-(4-Methoxyphenyl)-1,2,3,5-diselenadiazole

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Abstract

The analysis of the title compound, $C_8H_7N_2OSe_2$, shows that its crystals are composed of weakly linked dimers, with intramolecular Se—Se distances of 2.343 (3) and 2.345 (2) Å, intermolecular Se···Se distances of 3.193 (3) and 3.316 (3) Å, and lateral Se···Se interactions of 3.514 (2) and 3.579 (3) Å.

Comment

In the course of a detailed investigation of the chemical and electrochemical properties of neutral dithiadiazole (Boeré *et al.*, 1993) and diselenadiazole (Boeré, Moock & Parvez, 1994) radicals, we synthesized the title compound, (I). To complete the characterization of the selenium series of compounds and in order to understand its structure in the solid state, we undertook a single-crystal diffraction study of (I). Heterocycles of this type are important candidates for a new class of molecular metals based on even stacking of neutral 'π' radicals (Oakley, 1993).

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