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)
C3A†	0.5741 (7)	0.0533 (6)	0.1503 (16)	0.056 (3)
C4A†	0.6290 (5)	0.1941 (6)	0.2074 (13)	0.066 (3)
C5A†	0.6311 (7)	0.1570 (14)	−0.0377 (11)	0.114 (7)
C3B‡	0.5819 (6)	0.0856 (8)	0.2038 (10)	0.080 (3)
C4B‡	0.6415 (4)	0.2160 (4)	0.0880 (14)	0.083 (4)
C5B‡	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*.

We thank the Natural Sciences and Engineering Research Council (Canada) for providing the diffractometer through an equipment grant to the University of Calgary and for an operating grant to RTB. KHM is grateful to the A. von Humboldt Foundation (Germany) for the award of a Feodor–Lynen Fellowship. SDD was supported by the University of Lethbridge Research Fund.

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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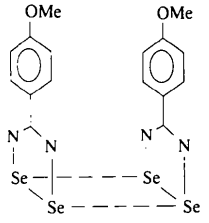
Abstract

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).



(I)

An *ORTEP*II (Johnson, 1976) illustration of the title compound is presented in Fig. 1 together with the atomic numbering scheme. Fig. 2 is a stereoview of the unit cell showing the molecular packing. The CN_2Se_2 and C_6H_4 rings are planar within experimental error. The two halves of the dimer are aligned almost parallel, being tilted apart by only 4.5 (5)°. The two methoxy groups within the dimer are directed in opposite directions, presumably in order to minimize steric repulsions, and are not involved in close interdimer contacts.

The two Se_2N_2C rings in the dimer, with a mean Se—Se bond length of 2.344 (3) Å, are joined by two non-equivalent interatomic Se···Se contacts of 3.193 (3) and 3.316 (3) Å. The dimers also exhibit lateral interdimer Se···Se interactions of 3.514 (2) and 3.579 (3) Å. There

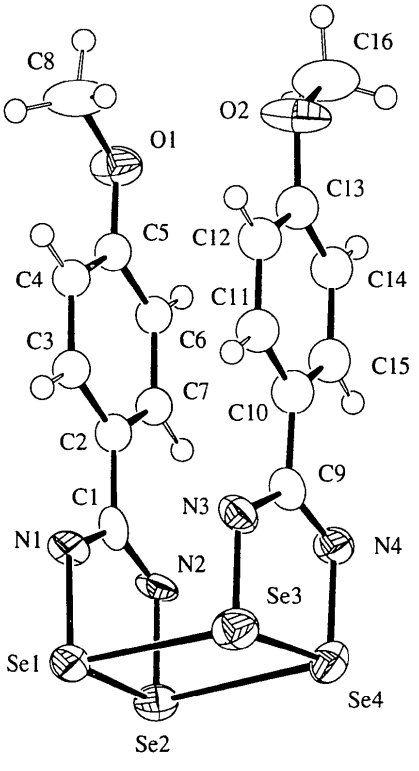


Fig. 1. A view of the title compound with the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% probability level. Phenyl C atoms are plotted as spheres and H atoms have been assigned arbitrary radii.

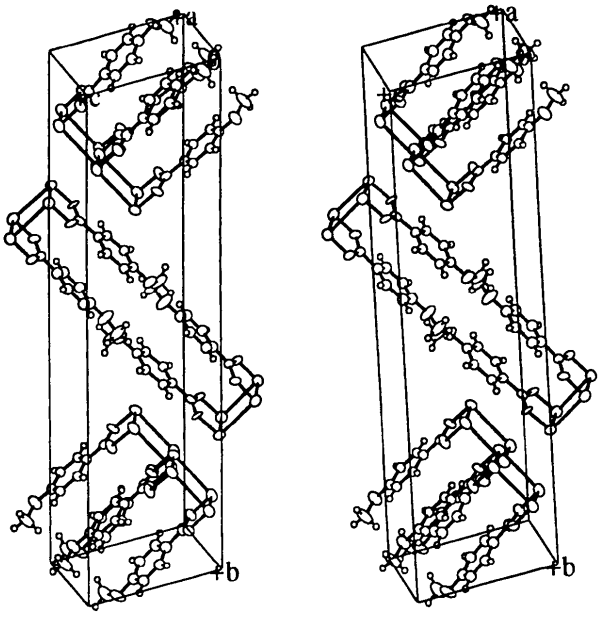


Fig. 2. A stereoview of the unit cell of the title compound showing the molecular packing.

are several Se···Se interactions in the range 3.870 (3)–4.159 (3) Å involving adjacent dimers. Secondary interactions of this type have been reported previously, *e.g.* for $(C_6H_5CN_2Se_2)_2$ (3.815 and 4.075 Å; Del Bel Belluz *et al.*, 1989), for the 2-, 3- and 4-cyano derivatives of $(PhCN_2Se_2)_2$ [3.829 (3)–4.066 (4) Å; Cordes, Haddon, Hicks, Oakley & Palstra, 1992] and for the 3,5-dicyano derivative of $(PhCN_2Se_2)_2$ [4.038 (2) and 4.079 (2) Å; Davis, Hicks, Oakley, Zhao & Taylor, 1993]. The interactions between the $(CN_2Se_2)_2$ pairs in the title compound, which pack the dimers together in a herringbone fashion, are strongly reminiscent of those in the structure of the bifunctional radical $[1,4\text{-}(Se_2N_2C)_2C_6H_4]_2$ (Cordes *et al.* 1991). The latter structure has two diselenadiazole units per monomer, so that herringbone interdimer interactions occur at both ends of the molecule. In contrast, the title compound contains alternating layers of interacting diselenadiazole moieties and weakly attracting methoxy groups.

Experimental

The title compound was prepared according to Boeré, Moock & Parvez (1994). Crystals were obtained as dark brown prisms by sublimation in a sealed Pyrex tube contained in a three-zone (423/353/303 K) tube furnace under static vacuum (10^{-2} Torr; 1 Torr = 133.322 Pa). The crystals were collected in the 353 K zone.

Crystal data

$C_8H_7N_2OSe_2$
M_r = 305.08

Mo $K\alpha$ radiation
λ = 0.71069 Å

Monoclinic
$P2_1/n$
$a = 6.155\,(1)$ Å
$b = 32.871\,(5)$ Å
$c = 9.270\,(1)$ Å
$\beta = 100.87\,(1)°$
$V = 1841.9\,(4)$ Å^3
$Z = 8$
$D_x = 2.20$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 10.0$–$20.0°$
$\mu = 7.988$ mm^{-1}
$T = 295$ K
Prism
0.50 × 0.25 × 0.20 mm
Dark brown

Data collection

AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.304$, $T_{max} = 0.999$
3659 measured reflections
3342 independent reflections
1332 observed reflections [$I > 3\sigma(I)$]

$R_{int} = 0.039$
$\theta_{max} = 25.0°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 39$
$l = -11 \rightarrow 10$
3 standard reflections monitored every 200 reflections
intensity decay: 1.59%

Refinement

Refinement on F
$R = 0.0530$
$wR = 0.0530$
$S = 2.070$
1332 reflections
142 parameters
H atoms riding
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{max} = 0.016$

$\Delta\rho_{max} = 0.90$ e Å^{-3}
$\Delta\rho_{min} = -0.85$ e Å^{-3}
Extinction correction: Zachariasen (1968) type 2, Gaussian isotropic
Extinction coefficient: 24.58852
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* (Å^2)

U_{iso} for phenyl C atoms; $U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for others.

	x	y	z	U_{eq}/U_{iso}
Se1	0.3437 (3)	0.25368 (6)	0.5266 (2)	0.0442 (7)
Se2	0.0265 (3)	0.25355 (6)	0.6327 (2)	0.0434 (7)
Se3	0.5766 (3)	0.18367 (7)	0.7401 (2)	0.0460 (7)
Se4	0.2634 (3)	0.17931 (7)	0.8489 (2)	0.0461 (7)
O1	−0.368 (2)	0.0866 (4)	−0.0079 (11)	0.050 (4)
O2	−0.019 (2)	0.0026 (4)	0.2239 (13)	0.064 (5)
N1	0.222 (2)	0.2168 (4)	0.3944 (12)	0.040 (5)
N2	−0.100 (2)	0.2162 (4)	0.5014 (13)	0.036 (5)
N3	0.472 (2)	0.1448 (4)	0.6153 (13)	0.038 (5)
N4	0.149 (2)	0.1400 (4)	0.7236 (13)	0.040 (5)
C1	0.022 (3)	0.2035 (5)	0.4035 (15)	0.034 (6)
C2	−0.082 (2)	0.1728 (3)	0.2938 (9)	0.033 (2)
C3	0.041 (1)	0.1578 (3)	0.1938 (11)	0.033 (2)
C4	−0.050 (2)	0.1287 (3)	0.0906 (9)	0.033 (2)
C5	−0.264 (2)	0.1146 (3)	0.0872 (8)	0.033 (2)
C6	−0.388 (1)	0.1296 (3)	0.1872 (11)	0.033 (2)
C7	−0.297 (2)	0.1587 (3)	0.2905 (9)	0.033 (2)
C8	−0.247 (3)	0.0696 (6)	−0.1089 (19)	0.072 (8)
C9	0.272 (3)	0.1287 (5)	0.6244 (15)	0.037 (6)
C10	0.191 (2)	0.0951 (3)	0.5235 (10)	0.043 (2)
C11	0.332 (1)	0.0784 (3)	0.4378 (11)	0.043 (2)
C12	0.257 (2)	0.0474 (3)	0.3381 (10)	0.043 (2)
C13	0.040 (2)	0.0330 (3)	0.3242 (10)	0.043 (2)
C14	−0.101 (1)	0.0497 (3)	0.4099 (11)	0.043 (2)
C15	−0.025 (2)	0.0807 (3)	0.5096 (10)	0.043 (2)
C16	−0.241 (3)	−0.0106 (7)	0.1960 (19)	0.072 (8)

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

Se1—Se2	2.345 (2)	Se2···Se4^i	3.579 (3)
Se1···Se3	3.193 (3)	Se1—N1	1.79 (1)
Se2···Se4	3.316 (3)	Se2—N2	1.80 (1)
Se3—Se4	2.343 (3)	Se3—N3	1.76 (1)
Se1···Se3^i	3.514 (2)	Se4—N4	1.79 (1)
Se2—Se1···Se3	92.9 (1)	Se4—Se3—N3	90.6 (5)
Se1—Se2···Se4	87.1 (1)	Se1···Se3—N3	92.9 (4)
Se1···Se3—Se4	90.1 (1)	Se3—Se4—N4	91.2 (4)
Se2···Se4—Se3	89.9 (1)	Se2···Se4—N4	93.7 (4)
Se2—Se1—N1	90.6 (4)	Se1—N1—C1	117 (1)
Se3···Se1—N1	91.0 (4)	Se2—N2—C1	116 (1)
Se1—Se2—N2	90.3 (4)	Se3—N3—C9	117 (1)
Se4···Se2—N2	89.5 (4)	Se4—N4—C9	115 (1)

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

The space group was uniquely determined from the systematic absences: $h0l$ when $h + l = 2n + 1$ and $0k0$ when $k = 2n + 1$. The structure was solved by direct methods and refined by full-matrix least-squares calculations. Non-H atoms, except for the C atoms of phenyl rings, were allowed anisotropic displacement parameters; phenyl C atoms were included as regular hexagons with overall isotropic displacement parameters. Allowance was made for anomalous dispersion (Ibers & Hamilton, 1964). All calculations were performed on a Silicon Graphics Personal Iris D/35 computer.

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: *TEXSAN*. 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: FG1061). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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8-Amino-7-(2-hydroxy-3-morpholinopropyl)theophylline

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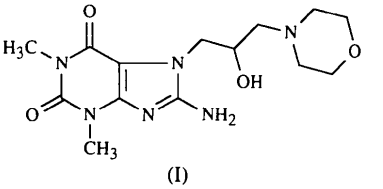
Abstract

The purine fused-ring skeleton in the title compound, 8-amino-7-(2-hydroxy-3-morpholinopropyl)-1,3-dimethyl-3,7-dihydro-1*H*-purine-2,6-dione, $C_{14}H_{22}N_6O_4$, is planar, while the morpholine ring adopts a chair conformation. Statistical disorder occurs within the hydroxy group. The structure is stabilized by a network of intermolecular hydrogen bonds and the conformation of the 7-amino-hydroxyalkyl substituent is determined by an O—H···N intramolecular hydrogen bond.

Comment

Pharmacological investigation of a series of 7,8-disubstituted derivatives of theophylline revealed that some of these compounds exhibited potent antihypertensive and vasodilator activity (Łucka-Sobstel *et al.*, 1985; Gorczyca, Pawłowski, Mrozikiewicz, Kozłowska & Wasik, 1986; Olejnik *et al.*, 1989). Of a number of compounds studied, the title structure, (I), was chosen for more detailed pharmacological screening (Pawłowski, Gorczyca, Bobkiewicz-Kozłowska, Chodera & Mrozikiewicz, 1991). Circulatory effects, mainly antihypertensive activity, the beneficial effect on cerebral blood-flow autoregulation (Kozłowska *et al.*, 1989) and low toxicity in comparison with its mother compound theophylline (aminophylline) suggest the need to test the clinical effectiveness of (I) in order to obtain a new therapeutic agent.



(I)

We report here the results of the X-ray structure determination of (I) as part of a larger structural and pharmacological study on 7,8-disubstituted theophylline derivatives. The complete crystal structure analysis was expected to yield information concerning the effects of substituents on receptor affinities of compound (I).

Bond lengths and angles in the theophylline skeleton do not differ significantly from those reported for theophylline (Sutor, 1958) and its 7,8-disubstituted derivatives (Karolak-Wojciechowska & Pawłowski, 1990; Karczmarzyk, Karolak-Wojciechowska & Pawłowski, 1991). In the purine fused-ring system the six-membered ring is planar to within 0.012 (3) Å and the five-membered ring is planar to within 0.004 (3) Å. These two rings are inclined at an angle of 0.9 (1)° with respect to each other. The length of the N6—C6 bond and the sum of the valence angles around the N6 atom (358.2°) show that this atom is sp^2 hybridized and that the amino group is conjugated with the π system of the imidazole ring.

The morpholine ring adopts a chair conformation with puckering parameters (Cremer & Pople, 1975) of Q = 0.5593 Å and θ = 178.4°. The most noteworthy feature is the geometry of the pyramidal N13 ring atom. The C12—N13—C18 angle is significantly larger than the C12—N13—C14 and C14—N13—C18 angles. The opening of this angle indicates a distorted tetrahedral configuration around the N13 atom, probably caused by steric effects. We were particularly interested in the conformation of the aminohydroxyalkyl substituent in the 7-position of the molecule. The torsion angles C6—N5—C10—C11 = −77.0 (3), N5—C10—C11—C12 = −73.2 (3), C10—C11—C12—N13 = −179.7 (3) and C11—C12—N13—C14 = 160.1 (3)° indicate a *gauche*–