N1—N2	1.423 (4)	C2'—C3'	1.520 (5)
N1—C5	1.373 (4)	C3'—C4'	1.516 (5)
N1—C1'	1.436 (4)	C4'—C5'	1.508 (5)
C1'—O6'—C5'	110.8 (2)	O6—C5—N1	124.6 (3)
N2—N1—C5	109.2 (3)	O6—C5—C4	127.8 (3)
N2—N1—C1'	119.1 (3)	N1—C5—C4	107.6 (3)
C5—N1—C1'	123.8 (3)	O6'—C1'—N1	108.2 (3)
N1—N2—C3	110.7 (3)	O6'—C1'—C2'	111.8 (3)
O7—C3—N2	124.9 (4)	N1—C1'—C2'	112.8 (3)
O7—C3—C4	126.4 (3)	C1'—C2'—C3'	109.8 (3)
N2—C3—C4	108.8 (3)	O7'—C3'—C2'	110.4 (3)
C3—C4—C5	102.1 (3)	O7'—C3'—C4'	110.6 (3)
C3—C4—C6	113.0 (3)	C2'—C3'—C4'	109.6 (3)
C3—C4—C7	108.2 (3)	O8'—C4'—C3'	112.3 (3)
C5—C4—C6	111.5 (3)	O8'—C4'—C5'	107.5 (3)
C5—C4—C7	110.3 (3)	C3'—C4'—C5'	108.8 (3)
C6—C4—C7	111.3 (3)	O6'—C5'—C4'	111.7 (3)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O7'ⁱ	0.89 (4)	1.88 (4)	2.746 (4)	163 (4)
O7'—H7'···O8'ⁱⁱ	0.98	1.83	2.763 (4)	157
O8'—H8'···O7'ⁱⁱⁱ	1.07	1.74	2.755 (5)	155
C5'—H5'a···O6'ⁱᵛ	0.95	2.55	3.471 (5)	165

Symmetry codes: (i) $\frac{3}{2}-x, -y, \frac{1}{2}+z$; (ii) $x, y, z-1$; (iii) $\frac{3}{2}-x, -y, z-\frac{1}{2}$; (iv) $\frac{1}{2}-x, -y, \frac{1}{2}+z$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *TEXSAN ORTEP* (Johnson, 1965). Software used to prepare material for publication: *TEXSAN FINISH*; *PLATON* (Spek, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1174). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-(p-Methoxyphenyl)-2-methyl-4-nitroimidazole and 1-(p-Acetylphenyl)-2-methyl-4-nitroimidazole

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Abstract

The title compounds, $C_{11}H_{11}N_3O_3$ and $C_{12}H_{11}N_3O_3$, are monomers. The angle between the planes of the phenyl and imidazole rings is 63.9 (6)° in the former compound and 51.8 (5)° in the latter. The nitro groups are twisted by 3.9 (5) and 5.1 (5)°, respectively, with respect to the imidazole rings.
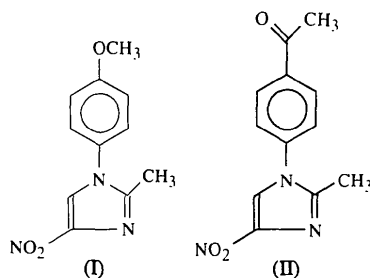
Comment

As part of a study of radiosensitizing nitroimidazoles (Suwiński & Salwińska, 1982) and a continuation of

studies presented recently (Kowalski, 1995), the crystal structures of 1-(p-methoxyphenyl)-2-methyl-4-nitroimidazole, (I), and 1-(p-acetylphenyl)-2-methyl-4-nitroimidazole, (II), have been determined.

1-Arylnitroimidazoles have been synthesized recently by Salwińska & Suwiński (1990). The two title compounds compounds do not have H atoms which are capable of forming hydrogen bonds. Neither the imidazole nor the phenyl rings are stacked. The aromaticity indexes I_5 (Bird, 1985) and HOMA (Gdaniec, Turowska-Tyrk & Krygowski, 1989) have values of 67.9 and 0.823, respectively, for unsubstitued imidazole [structural data for these calculations are from McMullan, Epstein, Ruble & Craven (1979), uncorrected for libration]. The title compounds have lower values of 58.5 and 0.490 for compound (I) and 60.6 and 0.627 for compound (II). The observed angles between the least-squares planes of the imidazole and phenyl rings are similar to those found in other 1-arylimidazoles (Ishihara et al., 1992).
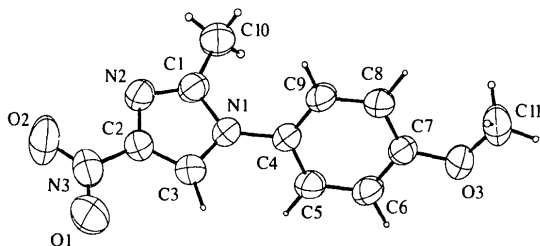

Fig. 1. The molecular structure and atom-numbering scheme of compound (I) shown with 50% probability ellipsoids.
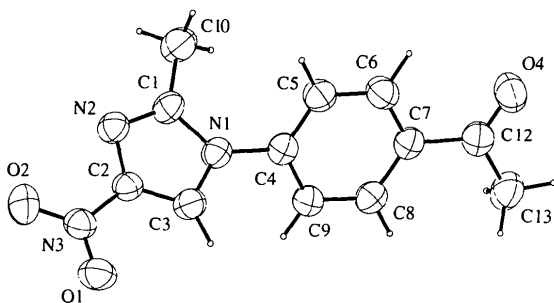

Fig. 2. The molecular structure and atom-numbering scheme of compound (II) shown with 50% probability ellipsoids.

Experimental

Compounds (I) and (II) were synthesized according to Salwińska & Suwiński (1990). Crystals were formed by slow evaporation from a water/ethanol solution.

Compound (I)

Crystal data

$C_{11}H_{11}N_3O_3$	Cu $K\alpha$ radiation
$M_r = 233.23$	$\lambda = 1.54178$ Å
Monoclinic	Cell parameters from 25
$P2_1/c$	reflections
$a = 12.299\,(2)$ Å	$\theta = 16-25°$
$b = 8.046\,(2)$ Å	$\mu = 0.849$ mm^{-1}
$c = 11.563\,(2)$ Å	$T = 293\,(2)$ K
$\beta = 90.83\,(3)°$	Irregular
$V = 1144.1\,(4)$ Å^3	$0.45 \times 0.30 \times 0.25$ mm
$Z = 4$	Colourless
$D_x = 1.354$ Mg m^{-3}	

Data collection

Kuma KM-4 automated four-circle κ-axis diffractometer	$R_{int} = 0.0105$
	$\theta_{max} = 79.92°$
$\omega/2\theta$ scans	$h = 15 \to 15$
Absorption correction:	$k = 0 \to 10$
none	$l = 0 \to 14$
2514 measured reflections	2 standard reflections
2385 independent reflections	monitored every 50
1614 observed reflections	reflections
$[I > 3\sigma(I)]$	intensity decay: 3.2%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = 0.630$
$R(F) = 0.0409$	$\Delta\rho_{max} = 0.113$ e Å^{-3}
$wR(F^2) = 0.1628$	$\Delta\rho_{min} = -0.122$ e Å^{-3}
$S = 1.120$	Extinction correction: none
2179 reflections	Atomic scattering factors
198 parameters	from *International Tables*
H atoms refined isotropically	*for Crystallography* (1992,
$w = 1/[\sigma^2(F_o^2) + (0.0763P)^2 + 0.0557P]$	Vol. C, Tables 4.2.6.8 and 6.1.1.4)
where $P = (F_o^2 + 2F_c^2)/3$	

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

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

	x	y	z	U_{eq}
O1	1.14314 (13)	0.3797 (3)	0.4787 (2)	0.1045 (6)
O2	1.13408 (14)	0.3013 (3)	0.6554 (2)	0.1052 (6)
O3	0.51503 (10)	0.1041 (2)	0.11819 (10)	0.0688 (4)
N1	0.84297 (10)	0.1669 (2)	0.45103 (10)	0.0557 (3)
N2	0.93710 (11)	0.1546 (2)	0.61565 (11)	0.0608 (4)
N3	1.09668 (12)	0.3102 (2)	0.55785 (15)	0.0707 (4)
C1	0.84631 (13)	0.1111 (2)	0.56381 (13)	0.0571 (4)
C2	0.99299 (11)	0.2388 (2)	0.53389 (13)	0.0578 (4)
C3	0.93842 (12)	0.2479 (2)	0.43074 (14)	0.0608 (4)
C4	0.75565 (12)	0.1476 (2)	0.36824 (12)	0.0535 (3)
C5	0.77232 (12)	0.0586 (2)	0.26773 (13)	0.0581 (4)
C6	0.68937 (13)	0.0464 (2)	0.18695 (13)	0.0605 (4)
C7	0.58958 (12)	0.1222 (2)	0.20567 (12)	0.0541 (3)

C8	0.57282 (12)	0.2099 (2)	0.30668 (13)	0.0604 (4)
C9	0.65666 (13)	0.2233 (2)	0.38762 (13)	0.0619 (4)
C10	0.7580 (2)	0.0118 (3)	0.6157 (2)	0.0752 (5)
C11	0.4145 (2)	0.1892 (4)	0.1276 (2)	0.0849 (6)

Compound (II)

Crystal data

$C_{12}H_{11}N_3O_3$
$M_r = 245.24$
Triclinic
$P\bar{1}$
$a = 7.4930$ (10) Å
$b = 8.217$ (2) Å
$c = 10.332$ (2) Å
$\alpha = 90.46$ (2)°
$\beta = 105.24$ (2)°
$\gamma = 110.59$ (2)°
$V = 571.0$ (2) Å^3
$Z = 2$
$D_x = 1.426$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 16–32°$
$\mu = 0.882$ mm^{-1}
$T = 293$ (2) K
Irregular
$0.40 \times 0.30 \times 0.28$ mm
Colourless

Data collection

Kuma KM-4 automated four-circle κ-axis diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2517 measured reflections
2344 independent reflections
1282 observed reflections
$[I > 2\sigma(I)]$

$R_{int} = 0.0105$
$\theta_{max} = 81.53°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 9$
$l = -12 \rightarrow 12$
2 standard reflections monitored every 50 reflections
intensity decay: 4%

Refinement

Refinement on F^2
$R(F) = 0.0424$
$wR(F^2) = 0.2912$
$S = 0.855$
1606 reflections
171 parameters
H atoms refined as rigid groups
$w = 1/[\sigma^2(F_o^2) + (0.1134P)^2 + 0.4209P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.013$

$\Delta\rho_{max} = 0.108$ e Å^{-3}
$\Delta\rho_{min} = -0.115$ e Å^{-3}
Extinction correction: SHELXL93
Extinction coefficient: 0.022 (4)
Atomic scattering factors from International Tables for Crystallography (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 2. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (II)

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

	x	y	z	U_{eq}
O1	0.5960 (3)	-0.2303 (3)	0.3401 (2)	0.0595 (6)
O2	0.4104 (3)	-0.3140 (3)	0.4735 (2)	0.0694 (7)
O4	0.2533 (3)	0.7331 (3)	-0.1047 (2)	0.0640 (6)
N1	0.2862 (3)	0.0915 (3)	0.2284 (2)	0.0428 (6)
N2	0.2342 (3)	-0.0779 (3)	0.3919 (2)	0.0470 (6)
N3	0.4643 (3)	-0.2233 (3)	0.3873 (2)	0.0482 (6)
C1	0.1851 (4)	0.0425 (3)	0.3250 (2)	0.0439 (6)
C2	0.3699 (3)	-0.1032 (3)	0.3379 (2)	0.0417 (6)
C3	0.4055 (4)	-0.0039 (3)	0.2371 (2)	0.0457 (7)
C4	0.2670 (3)	0.2144 (3)	0.1324 (2)	0.0419 (6)
C5	0.2885 (4)	0.3811 (3)	0.1769 (3)	0.0512 (7)

C6	0.2687 (4)	0.4975 (4)	0.0835 (3)	0.0503 (7)
C7	0.2299 (3)	0.4494 (3)	-0.0527 (2)	0.0436 (6)
C8	0.2114 (4)	0.2816 (3)	-0.0949 (3)	0.0480 (7)
C9	0.2294 (4)	0.1635 (4)	-0.0023 (3)	0.0470 (7)
C10	0.0423 (4)	0.1177 (4)	0.3480 (3)	0.0593 (8)
C12	0.2179 (4)	0.5830 (4)	-0.1489 (3)	0.0491 (7)
C13	0.1649 (5)	0.5318 (4)	-0.2971 (3)	0.0630 (8)

Table 3. Selected interatomic distances (Å) and bond angles (°) for compounds (I) and (II)

	(I)	(II)		(I)	(II)
O1—N3	1.221 (2)	1.228 (3)	C1—C10	1.482 (3)	1.476 (4)
O2—N3	1.214 (2)	1.225 (3)	C2—C3	1.362 (2)	1.352 (3)
O3—C7	1.363 (2)	–	C4—C5	1.383 (2)	1.382 (3)
O3—C11	1.419 (3)	–	C4—C9	1.382 (2)	1.376 (3)
O4—C12	–	1.224 (3)	C5—C6	1.377 (2)	1.381 (4)
N1—C3	1.366 (2)	1.368 (3)	C6—C7	1.390 (2)	1.384 (4)
N1—C1	1.379 (2)	1.386 (3)	C7—C8	1.383 (2)	1.390 (4)
N1—C4	1.437 (2)	1.437 (3)	C7—C12	–	1.497 (4)
N2—C1	1.307 (2)	1.314 (3)	C8—C9	1.387 (2)	1.384 (4)
N2—C2	1.358 (2)	1.354 (3)	C12—C13	–	1.494 (4)
N3—C2	1.423 (2)	1.430 (3)			

	(I)	(II)
C7—O3—C11	117.98 (14)	–
C3—N1—C1	107.69 (12)	107.3 (2)
C3—N1—C4	124.97 (12)	125.3 (2)
C1—N1—C4	127.34 (13)	127.3 (2)
C1—N2—C2	104.58 (13)	104.3 (2)
O1—N3—O2	123.2 (2)	123.2 (2)
O2—N3—C2	118.9 (2)	119.0 (2)
O1—N3—C2	117.8 (2)	117.8 (2)
N2—C1—N1	111.10 (14)	110.9 (2)
N2—C1—C10	125.8 (2)	125.4 (2)
N1—C1—C10	123.13 (15)	123.7 (2)
N2—C2—C3	112.87 (13)	113.5 (2)
N2—C2—N3	121.84 (14)	121.4 (2)
C3—C2—N3	125.29 (15)	125.1 (2)
C2—C3—N1	103.75 (13)	103.9 (2)
C5—C4—C9	120.39 (13)	121.3 (2)
C5—C4—N1	119.83 (13)	119.7 (2)
C9—C4—N1	119.74 (13)	119.0 (2)
C6—C5—C4	119.32 (14)	119.0 (2)
C5—C6—C7	120.68 (13)	120.8 (2)
O3—C7—C8	125.06 (13)	–
O3—C7—C6	115.05 (13)	–
C6—C7—C12	–	118.5 (2)
C8—C7—C12	–	122.3 (2)
C8—C7—C6	119.88 (13)	119.1 (2)
C7—C8—C9	119.42 (13)	120.5 (2)
C4—C9—C8	120.29 (13)	119.2 (2)
O4—C12—C13	–	120.9 (2)
O4—C12—C7	–	119.4 (2)
C13—C12—C7	–	119.6 (2)

The unit-cell parameters and space groups were found from oscillation and Weissenberg photographs, and confirmed by preliminary diffractometer data collection. All non-H atom positions were found by direct methods and refined. All H-atom positions were calculated geometrically and refined with isotropic displacement parameters for compound (I), and as rigid groups using the AFIX card of SHELXL93 (Sheldrick, 1993) with the isotropic displacement factors set equal to $1.5U_{eq}$ that of the parent atom for methyl-H atoms and $1.2U_{eq}$ for other H atoms for compound (II).

For both compounds, data collection: Kuma KM-4 User's Guide (Kuma, 1989); program(s) used to solve structures: SHELXS86 (Sheldrick, 1990); molecular graphics: ORTEPII (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1279). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,3-Dihydro-1,4-dithiaphenanthrene 1,4-Dioxide

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Abstract

The crystal and molecular structure of the title compound, $C_{12}H_{10}O_2S_2$, has been determined. The most relevant features are the *trans* disposition of the sulfinyl groups and the distorted 1,3-diplanar form of the 1,4-dithiane 1,4-dioxide ring.

Comment

As part of an investigation of the synthesis of organic compounds having possible pharmacological activity (Plumet, 1995), 2,3-dihydro-1,4-dithiaphenanthrene 1,4-

dioxide, (I), was obtained and its structure analyzed by standard analytical techniques (microanalysis, MS, NMR and IR). In order to obtain information about the stereochemistry of the sulfinyl groups in the 1,4-dithiane 1,4-dioxide ring and to confirm the assigned structure, the X-ray analysis of (I) was undertaken.

(I)
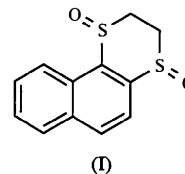
The structure of (I) consists of an essentially planar naphthalene ring condensed with a 1,4-dithiane 1,4-dioxide ring. The S atoms lie only 0.166 (2) and 0.202 (2) Å from the plane of the aromatic ring. Bond lengths and angles are in good agreement with those found in analogous compounds, in spite of the possible disorder of the C2 atom; the crystallographic data used were retrieved from the Cambridge Structural Database (Allen *et al.*, 1979). According to the data so far accumulated (Allen *et al.*, 1987), a normal S—C single-bond length is about 1.79 (1) Å. The C—S distances found in the present compound [1.772 (7), 1.802 (4), 1.782 (4) and 1.795 (5) Å] imply a mostly single-bond character and consequently there is no evidence of conjugation between the naphthalene ring and the sulfinyl groups. The S=O bond lengths of 1.467 (4) and 1.506 (4) Å are very close to the corresponding double-bond length of 1.497 (13) Å found by Allen *et al.* (1987). The C—S—C angles of 100.8 (3) and 96.7 (3)° are also typical.
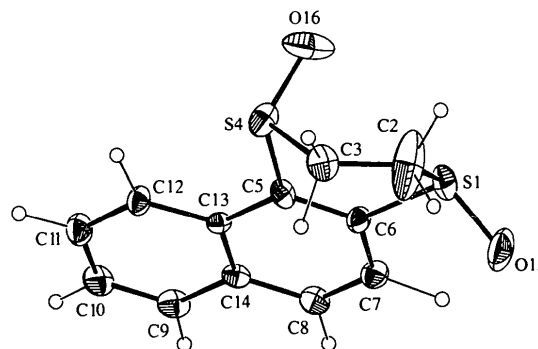

Fig. 1. The structure of the title compound showing 30% probability displacement ellipsoids.

The 1,4-dithiane 1,4-dioxide ring has a distorted 1,3-diplanar form (Toromanoff, 1980) as may be deduced from the torsion angles C2—S1—C6—C5 36.3 (5), C6—S1—C2—C3 −14.8 (10), S1—C2—C3—S4 −35.9 (11), C2—C3—S4—C5 64.8 (7), C3—S4—C5—C6 −46.8 (4) and S4—C5—C6—S1 −2.0 (6)°. The sulfinyl groups were found to be mutually *trans* in this ring.