*Data collection*

Rigaku AFC-5*R* diffractometer
ω scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.77$, $T_{\max} = 1.05$
7235 measured reflections
7084 independent reflections
4201 observed reflections [$I > 3\sigma(I)$]

$R_{\text{int}} = 0.017$
$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 14$
$k = -1 \rightarrow 52$
$l = -1 \rightarrow 14$
3 standard reflections monitored every 150 reflections
intensity variation: insignificant

*Refinement*

Refinement on *F*
$R = 0.0516$
$wR = 0.0479$
$S = 2.125$
4201 reflections
595 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + (0.005F_o)^2]$
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.40$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.30$ e Å$^{-3}$

Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
Absolute configuration: assigned to agree with the known chirality at C(5) as established by the synthesis of the compound

During the refinement the behaviour of some of the acetyl groups indicated that they are slightly disordered; however, a disordered model could not be refined satisfactorily. An ordered model was employed for the final refinement and the enlarged thermal ellipsoids for some of the atoms of these groups reflect the 'smeared out' electron density resulting from the disorder. All of the H atoms were placed in geometrically calculated positions and were assigned fixed isotropic displacement parameters with values of 1.2 × $U_{\text{eq}}$ of the parent C atom. Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: direct methods in *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN FINISH*.

The National University of Singapore is thanked for financial support.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71833 (52 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1081]

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# 1-Methyl-2-methylthio-5-nitroimidazole, $C_5H_7N_3O_2S$

Tomasz A. Olszak, Oswald M. Peeters,
Norbert M. Blaton and Camiel J. De Ranter

*Laboratorium voor Analytische Chemie en
Medicinale Fysicochemie, Faculteit Farmaceutische
Wetenschappen, Katholieke Universiteit Leuven,
Van Evenstraat 4, B-3000 Leuven, Belgium*

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## Abstract

In the title molecule, the imidazole ring is almost coplanar with the neighbouring attached atoms.

## Comment

Investigations of nitroimidazole derivatives have been performed in order to analyse the structure–activity relationships of these compounds, which are applied worldwide as drugs, either as radiosensitizers or antiprotozoic and antibiotic agents.

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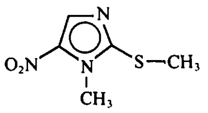
The crystal of the title compound (I) contains well ordered molecules of 1-methyl-2-methylthio-5-nitroimidazole. The imidazole ring is planar. The maximum deviation from the weighted least-squares plane through the non-H atoms is 0.002 (2) Å; atoms attached to the ring are almost in the ring plane and have a maximum deviation of 0.024 (1) Å. The dihedral angle between the ring and the nitro group is 179.0 (2)°. The geometry of the five-membered ring together with the attached atoms is in good agreement, to within 0.04 Å for distances and 2.5° for angles, with that of the closest structure possessing a tri-substituted imidazole ring (Dickens *et al.*, 1981).



(I)

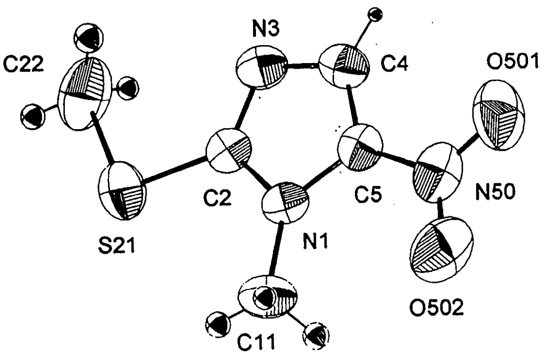


Fig. 1. A perspective view of the molecule with atomic numbering scheme. The displacement ellipsoids are plotted at the 50% probability level.

## Experimental

### *Crystal data*

$C_5H_7N_3O_2S$
$M_r$ = 173.19
Monoclinic
$P2_1/n$
$a$ = 7.769 (1) Å
$b$ = 6.575 (1) Å
$c$ = 15.258 (3) Å
$\beta$ = 95.49 (4)°
$V$ = 775.8 (2) Å$^3$
$Z$ = 4
$D_x$ = 1.4827 Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda$ = 0.71069 Å
Cell parameters from 42 reflections
$\theta$ = 10.13–19.36°
$\mu$ = 0.354 mm$^{-1}$
$T$ = 293 K
Block
0.3 × 0.3 × 0.4 mm
Yellow
Crystal source: grown from 3:1 ethanol/acetonitrile solution

### *Data collection*

Stoe Stadi-4 four-circle diffractometer
$\omega$ scans
Absorption correction: none
4071 measured reflections
1794 independent reflections
1408 observed reflections [$I > 2.0\sigma(I)$]

$R_{\rm int}$ = 0.011
$\theta_{\rm max}$ = 27.50°
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 9$
$l = -20 \rightarrow 20$
3 standard reflections
frequency: 60 min
intensity variation: 25%

### *Refinement*

Refinement on $F$
$R$ = 0.038
$wR$ = 0.062
$S$ = 0.962
1408 reflections
128 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.003802F^2]$

$(\Delta/\sigma)_{\rm max}$ = 0.006
$\Delta\rho_{\rm max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{\rm min}$ = −0.25 e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *CRYSRULER* (Rizzoli, Sangermano, Calestani & Andreetti, 1989)

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

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

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| N1 | 0.7351 (1) | 0.4436 (2) | 1.0391 (1) | 0.0416 (4) |
| C11 | 0.6811 (3) | 0.3077 (3) | 1.1071 (1) | 0.0609 (7) |
| C2 | 0.7124 (2) | 0.4085 (2) | 0.9514 (1) | 0.0427 (4) |
| S21 | 0.6076 (1) | 0.1919 (1) | 0.90871 (3) | 0.0620 (2) |
| C22 | 0.6282 (4) | 0.2302 (6) | 0.7937 (2) | 0.088 (1) |
| N3 | 0.7753 (2) | 0.5567 (2) | 0.9044 (1) | 0.053 (4) |
| C4 | 0.8424 (2) | 0.6936 (3) | 0.9639 (1) | 0.051 (5) |
| C5 | 0.8191 (2) | 0.6287 (3) | 1.0469 (1) | 0.044 (4) |
| N50 | 0.8703 (2) | 0.7282 (3) | 1.1269 (1) | 0.060 (6) |
| O501 | 0.9422 (2) | 0.8936 (3) | 1.1230 (1) | 0.086 (7) |
| O502 | 0.8417 (3) | 0.6468 (4) | 1.1961 (1) | 0.090 (6) |

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

| | | | |
|---|---|---|---|
| N1—C11 | 1.461 (2) | N3—C4 | 1.347 (2) |
| N1—C2 | 1.353 (2) | C4—C5 | 1.365 (2) |
| N1—C5 | 1.381 (2) | C5—N50 | 1.408 (2) |
| C2—S21 | 1.736 (1) | N50—O501 | 1.227 (2) |
| C2—N3 | 1.330 (2) | N50—O502 | 1.223 (2) |
| S21—C22 | 1.795 (3) | | |
| C2—N1—C5 | 104.6 (1) | N3—C4—C5 | 109.8 (2) |
| C11—N1—C5 | 130.1 (2) | N1—C5—C4 | 107.4 (2) |
| C11—N1—C2 | 125.4 (1) | C4—C5—N50 | 127.4 (2) |
| N1—C2—N3 | 112.9 (1) | N1—C5—N50 | 125.2 (2) |
| N1—C2—S21 | 121.6 (1) | C5—N50—O502 | 119.0 (2) |
| S21—C2—N3 | 125.6 (1) | C5—N50—O501 | 117.5 (2) |
| C2—S21—C22 | 99.8 (1) | O501—N50—O502 | 123.4 (2) |
| C2—N3—C4 | 105.3 (1) | | |

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1068). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# 2-(5-Nitro-2-styryl-1-imidazolyl)ethanol

Tomasz A. Olszak, Oswald M. Peeters,
Norbert M. Blaton and Camiel J. De Ranter

*Laboratorium voor Analytische Chemie en
Medicinale Fysicochemie, Faculteit Farmaceutische
Wetenschappen, Katholieke Universiteit Leuven,
Van Evenstraat 4, B-3000 Leuven, Belgium*

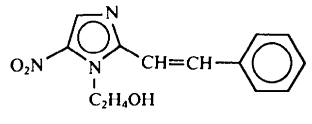
(*Received* 22 *September* 1993; *accepted* 8 *November* 1993)

## Abstract

The molecules of the title compound, $C_{13}H_{13}N_3O_3$, are linked in chains through hydrogen bonds along the *a* direction.

## Comment

The perspective view of the title compound (I) showing the atom-numbering scheme and hydrogen bonding is given in Fig. 1. The crystal contains well ordered molecules of 2-(5-nitro-2-styryl-1-imidazolyl)ethanol in the form previously observed in crystals of 2-(2-methyl-5-nitro-1-imidazolyl)ethanol (metronidazole) (Blaton, Peeters & De Ranter, 1979). The bond lengths and angles are in good agreement to within 0.005 Å and 1.6°, respectively, with those observed in metronidazole. More significant deviations are observed for C2—N3 (longer by 0.012 Å) and C2—C21 (shorter by 0.027 Å). These differences are undeniably a result of the conjugation effect between the C2—C21 single bond and the neighbouring C2=N3 and C21=C22 double bonds.



(I)

The five-membered ring is planar within 0.003 (2) Å. The nitro-group plane crosses the weighted least-squares imidazole plane at an angle of 4.7 (1)°. The benzene ring is planar and its weighted least-squares plane forms a dihedral angle of 5.7 (1)° with the imidazole plane. Although the molecule was expected to be planar except for the ethanol moiety, twisting is observed around the C2—C21 bond [N3—C2—C21—C22 21.9 (3)°]. This is probably caused by the molecular crowding or packing effects.

The molecules of the title compound are linked through O13—H13···N3$^i$ hydrogen bonds [1.99 (3) Å, 166 (2)°; symmetry code: (i) $x - 1, y, z$] forming chains along the *a* direction. Although for both the title compound and metronidazole (Blaton *et al.*, 1979) the same atoms are engaged in hydrogen bonding, the hydrogen-bonding patterns are different. This results in significant differences in the orientation of the ethanol moiety. The values of the torsion angle C5—N1—C11—C12 for the title molecule and metronidazole (Blaton *et al.*, 1979) are −106.8 (2) and 82.6 (2)°, respectively.

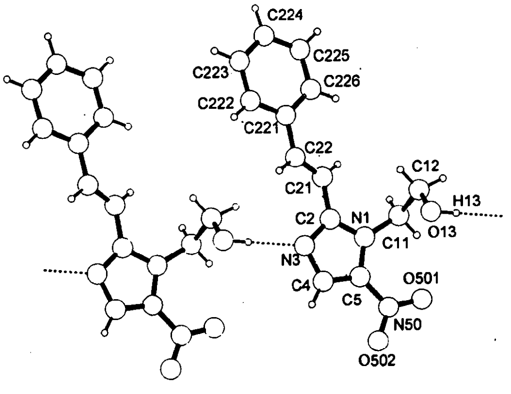


Fig. 1. The atomic numbering scheme and hydrogen bonding.

## Experimental

*Crystal data*

$C_{13}H_{13}N_3O_3$
$M_r = 259.26$
Monoclinic
$P2_1/c$
$a = 7.088\ (1)$ Å
$b = 7.116\ (1)$ Å
$c = 24.935\ (3)$ Å
$\beta = 95.03\ (1)°$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 40 reflections
$\theta = 10.38-12.48°$
$\mu = 0.094\ \text{mm}^{-1}$
$T = 293$ K
Prism