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# 1-(2-Chloroethyl)-2-methylsulfanyl-5-nitro-1*H*-benzimidazole

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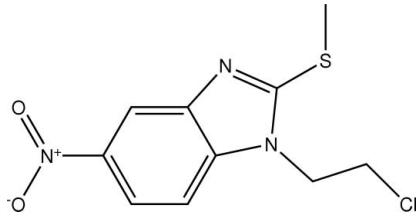
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.056; $wR$ factor = 0.115; data-to-parameter ratio = 22.4.

The title compound, $C_{10}H_{10}ClN_3O_2S$, exhibits three ring motifs, *viz.* $R_2^2(10)$, $R_2^2(8)$ and $S(5)$, arising from C—H···O, C—H···N and C—H···S interactions, respectvely, generating an [001] chain. A C—H···$\pi$ interaction (H···$Cg$ = 2.90 Å) is also present.

## Related literature

For background, see: Küçükbay *et al.* (2003); Bernstein *et al.* (1995).




## Experimental

### Crystal data

$C_{10}H_{10}ClN_3O_2S$
$M_r$ = 271.73
Triclinic, $P\overline{1}$
$a$ = 4.8564 (5) Å
$b$ = 10.224 (2) Å
$c$ = 12.464 (1) Å
$\alpha$ = 83.454 (5)°
$\beta$ = 79.372 (5)°
$\gamma$ = 79.719 (8)°
$V$ = 596.37 (14) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.49 mm$^{-1}$
$T$ = 294 K
0.50 × 0.25 × 0.20 mm

### Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
11977 measured reflections
3479 independent reflections
2256 reflections with $I > 2.0\sigma(I)$
$R_{int}$ = 0.052

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.115
$S$ = 0.92
3455 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.62 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

*Cg* is the centroid of the benzene ring.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C3—H3···O16$^{i}$ | 0.93 | 2.41 | 3.326 (4) | 168 |
| C6—H6···N7$^{ii}$ | 0.93 | 2.57 | 3.499 (4) | 175 |
| C11—H112···O17$^{iii}$ | 0.96 | 2.55 | 3.437 (4) | 154 |
| C10—H102···S13 | 0.96 | 2.68 | 3.168 (3) | 112 |
| C14—H142···$Cg^{iv}$ | 0.96 | 2.90 | 3.836 (4) | 165 |

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

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *CRYSTALS*.

We thank the Laboratoire de Physique des Interactions Ioniques and Spectropôle, Université de Provence, and the Université Paul Cézanne, Faculté des Sciences et Techniques de Saint Jérôme, Marseille, France, for the use of their diffractometer. We also thank Mr Abou Akoun for help and advice.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2655).

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o4798 [ **doi:10.1107/S1600536807058254** ]

# 1-(2-Chloroethyl)-2-methylsulfanyl-5-nitro-1*H*-benzimidazole

**S. D. Goulizan Bi, R. Kakou-Yao, T. G. Manh, A. J. Tenon and J. P. Aycard**

## Comment

Benzimidazole and its derivatives exhibit versatile pharmacological activities (*e.g.* Küçükbay *et al.*, 2003). In order to study the influence of new substituents on the activity of the benzimidazole derivative, the title compound, (I), has been synthesized and its structure has been determined (Fig. 1).

In this structure, the nine-membered benzimidazole ring system C1/N2/C3/C4/C5/C6/N7/C8/C9 is essentially planar, the maximum deviation from planarity being 0.013 (2) Å for atom N2. The packing can be expressed as a stacking of sheets running along the *c* axis. The sheets consist of a two-dimensional hydrogen bonding network (Fig. 2), which is described by the rings of graph-set motif $R_2^2(8)$, $R_2^2(10)$ and S(5) (Bernstein *et al.*, 1995). The supramolecular aggregation is completed by the presence of C—H···Π interaction between an H atom of the methyl groupe i.e H142 and the benzene ring at the symmetry position (2 − *x*, 2 − *y*, −*z*).

## Experimental

2-Methylthio-5-Nitro-1*H*-benzimidazole (1 g, 4.8 mmol), 1,2-dichloroethane (0.75 ml, 9.6 mmol), potassium carbonate (1.32 g, 9.6 mmol) and dry dimethylformamide (DMF) (10 ml) was stirred together at 349 K. After one day (24 h), water (50 ml) was added, and the products were extracted with ethylacetate (2 × 100 ml). The combined organics extracts were washed with brine (2 × 50 ml), dried (MgSO4) and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel. Elution: hexane/ethylacetate (80:20, *v*/*v*) to give the title compound as a yellow powder (0.55 g) with a melting point 401 K and 1,2-Bis(2-methylthio-5-Nitro-1*H*-benzimidazol-1-yl)ethane (0.5 g). The yellow powder of (I) was dissolved in acetone/pentane and after one day, yellow blocks of (I) were obtained. $^1$H NMR (DMSO, 300 MHz, p.p.m): δ: 2.7 (S, 3H, $CH_3$), 4,1 (t, 2H, $CH_2Cl$), 4.55 and 4.65 (2 t, 2H, $CH_2N$), 7.5–8.6 (m, 3H, $C_6H_3$). $^{13}$C NMR (DMSO, 300 MHz, p.p.m): δ: 14.46($CH_3$), 43 ($CH_2Cl$), 46 ($CH_2N$), 106.63, 110.11, 113.10, 142, 142.55, 147.32 (C4, C5, C6, C7, C8, C9), 159.40 (C═N).

## Refinement

All hydrogen atoms were placed at calculated positions, with C—H = 0.93Å (aromatic) or 0.96Å (methylene and methyle) and refined using a riding model with $U_{iso}$(H) = 1.2$U_{eq}$(*c*).

## Figures

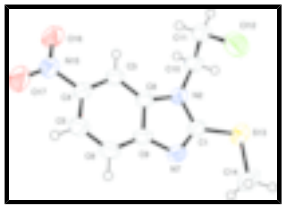

Fig. 1. The molecular structure of (I) showing displacement ellipsoids at the 50% probability level. H atoms are shown as spheres of arbitary radius.

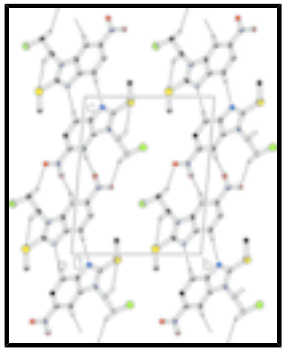

Fig. 2. Crystal packing of compound (I) viewed down the *a* axis, showing the rings of graph-set motif $R_2^2(8)$, $R_2^2(10)$ and S(5). Dashed lines indicate C—H···O, C—H···N and C—H···S hydrogen bonds (details are given in table 1). H atoms not involved in hydrogen bonds have been omitted for the clarity.

### 1-(2-Chloroethyl)-2-methylsulfanyl-5-nitro-1*H*-benzimidazole

*Crystal data*

| | |
|---|---|
| $C_{10}H_{10}Cl_1N_3O_2S_1$ | $Z = 2$ |
| $M_r = 271.73$ | $F_{000} = 280$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.513$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Melting point: 401 K |
| $a = 4.8564$ (5) Å | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $b = 10.224$ (2) Å | Cell parameters from 11977 reflections |
| $c = 12.464$ (1) Å | $\theta = 1.7$–$30.2$º |
| $\alpha = 83.454$ (5)º | $\mu = 0.49$ mm$^{-1}$ |
| $\beta = 79.372$ (5)º | $T = 294$ K |
| $\gamma = 79.719$ (8)º | Block, yellow |
| $V = 596.37$ (14) Å$^3$ | 0.50 × 0.25 × 0.20 mm |

*Data collection*

| | |
|---|---|
| Nonius KappaCCD diffractometer | 2256 reflections with $I > 2.0\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int} = 0.052$ |
| Monochromator: graphite | $\theta_{max} = 30.2$º |
| $T = 294$ K | $\theta_{min} = 1.7$º |
| φ scans | $h = -6 \rightarrow 6$ |
| Absorption correction: none | $k = -13 \rightarrow 14$ |
| 11977 measured reflections | $l = -17 \rightarrow 17$ |
| 3479 independent reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.056$ | H-atom parameters constrained |
| $wR(F^2) = 0.115$ | Method, part 1, Chebychev polynomial, (Watkin, 1994, *P*rince, 1982) [*w*eight] = 1.0/[$A_0$*$T_0$(x) + $A_1$*$T_1$(x) ··· + $A_{n-1}$]*$T_{n-1}$(x)] where $A_i$ are the Chebychev coefficients listed below and x = *F* /*F*max Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta*F*/6*sigma*F*)$^2$]$^2$ $A_i$ are: 203. 275. 116. 22.5 |
| $S = 0.92$ | $(\Delta/\sigma)_{max} = 0.0002$ |
| 3455 reflections | $\Delta\rho_{max} = 0.56$ e Å$^{-3}$ |
| 154 parameters | $\Delta\rho_{min} = -0.62$ e Å$^{-3}$ |
| 40 constraints | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. The 24 reflections (0 1 0, 0 2 0, 6 6 0, 0 13 0, 0 − 1 1, 0 0 1, 0 1 1, 0 0 2, 0 1 2, 0 1 3, 2 3 3, 2 2 4, −5 6 5, 3 5 6, 1 13 6, −3 − 10 8, 4 − 8 8, −4 − 6 8, −3 − 8 10, 6 3 10, −2 − 7 13, 2 − 3 15, 3 0 15, −1 3 15) have been measured with too low intensities. It might be caused by some systematical error, probably by shielding by a beam stop of these diffractions. They were not used in the refinement.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| H3 | 0.9516 | 0.9537 | 0.3842 | 0.0552* |
| H5 | 0.3229 | 1.1952 | 0.2531 | 0.0638* |
| H6 | 0.4150 | 1.0751 | 0.0989 | 0.0624* |
| C1 | 1.0486 (7) | 0.7740 (3) | 0.1034 (2) | 0.0484 |
| N2 | 1.0931 (5) | 0.7968 (2) | 0.20479 (18) | 0.0451 |
| C3 | 0.8469 (6) | 0.9773 (3) | 0.3276 (2) | 0.0458 |
| C4 | 0.6300 (7) | 1.0843 (3) | 0.3293 (2) | 0.0493 |
| C5 | 0.4680 (6) | 1.1222 (3) | 0.2467 (2) | 0.0535 |
| C6 | 0.5217 (7) | 1.0513 (3) | 0.1549 (2) | 0.0524 |
| N7 | 0.8409 (5) | 0.8575 (2) | 0.06702 (18) | 0.0496 |
| C8 | 0.8969 (6) | 0.9070 (3) | 0.2358 (2) | 0.0416 |
| C9 | 0.7415 (6) | 0.9436 (3) | 0.1493 (2) | 0.0438 |
| C10 | 1.2920 (6) | 0.7179 (3) | 0.2707 (2) | 0.0513 |

| | | | | |
|---|---|---|---|---|
| C11 | 1.1513 (7) | 0.6340 (3) | 0.3652 (2) | 0.0580 |
| Cl12 | 0.9721 (2) | 0.51698 (9) | 0.32109 (9) | 0.0798 |
| S13 | 1.2621 (2) | 0.64420 (9) | 0.03348 (7) | 0.0667 |
| C14 | 1.1046 (9) | 0.6618 (4) | −0.0875 (3) | 0.0722 |
| N15 | 0.5685 (7) | 1.1634 (3) | 0.4244 (2) | 0.0666 |
| O16 | 0.7024 (6) | 1.1302 (3) | 0.49822 (19) | 0.0857 |
| O17 | 0.3844 (8) | 1.2587 (3) | 0.4272 (2) | 0.1390 |
| H141 | 1.2031 | 0.5951 | −0.1354 | 0.0864* |
| H142 | 1.1169 | 0.7488 | −0.1244 | 0.0864* |
| H143 | 0.9088 | 0.6510 | −0.0675 | 0.0864* |
| H101 | 1.3873 | 0.7774 | 0.2993 | 0.0612* |
| H102 | 1.4284 | 0.6599 | 0.2244 | 0.0612* |
| H111 | 1.2943 | 0.5859 | 0.4052 | 0.0696* |
| H112 | 1.0163 | 0.6917 | 0.4123 | 0.0696* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0641 (19) | 0.0457 (15) | 0.0402 (14) | −0.0146 (14) | −0.0149 (13) | −0.0044 (12) |
| N2 | 0.0521 (14) | 0.0466 (13) | 0.0392 (12) | −0.0039 (11) | −0.0160 (10) | −0.0074 (10) |
| C3 | 0.0485 (16) | 0.0531 (16) | 0.0378 (14) | −0.0059 (13) | −0.0144 (12) | −0.0039 (12) |
| C4 | 0.0551 (18) | 0.0547 (17) | 0.0372 (14) | −0.0022 (14) | −0.0101 (13) | −0.0069 (12) |
| C5 | 0.0521 (18) | 0.0567 (18) | 0.0492 (16) | 0.0011 (14) | −0.0145 (14) | −0.0001 (14) |
| C6 | 0.0574 (18) | 0.0581 (18) | 0.0439 (15) | −0.0061 (15) | −0.0218 (14) | 0.0030 (13) |
| N7 | 0.0645 (16) | 0.0502 (13) | 0.0396 (12) | −0.0130 (12) | −0.0197 (11) | −0.0032 (10) |
| C8 | 0.0440 (15) | 0.0448 (14) | 0.0384 (13) | −0.0080 (12) | −0.0137 (11) | −0.0016 (11) |
| C9 | 0.0499 (16) | 0.0484 (15) | 0.0370 (13) | −0.0131 (13) | −0.0152 (12) | 0.0008 (11) |
| C10 | 0.0493 (17) | 0.0586 (18) | 0.0482 (16) | −0.0001 (14) | −0.0194 (13) | −0.0089 (13) |
| C11 | 0.066 (2) | 0.0572 (18) | 0.0492 (17) | 0.0041 (16) | −0.0207 (15) | −0.0033 (14) |
| Cl12 | 0.0984 (8) | 0.0616 (5) | 0.0847 (7) | −0.0180 (5) | −0.0308 (6) | 0.0057 (5) |
| S13 | 0.0882 (7) | 0.0606 (5) | 0.0533 (5) | −0.0004 (5) | −0.0189 (4) | −0.0199 (4) |
| C14 | 0.095 (3) | 0.083 (3) | 0.0470 (18) | −0.029 (2) | −0.0134 (18) | −0.0185 (17) |
| N15 | 0.075 (2) | 0.0695 (19) | 0.0499 (16) | 0.0137 (15) | −0.0149 (14) | −0.0143 (14) |
| O16 | 0.107 (2) | 0.0924 (19) | 0.0611 (15) | 0.0189 (16) | −0.0404 (15) | −0.0319 (14) |
| O17 | 0.167 (3) | 0.142 (3) | 0.086 (2) | 0.098 (3) | −0.052 (2) | −0.056 (2) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| H3—C3 | 0.930 | N7—C9 | 1.389 (3) |
| H5—C5 | 0.930 | C8—C9 | 1.407 (3) |
| H6—C6 | 0.930 | C10—C11 | 1.508 (4) |
| C1—N2 | 1.372 (3) | C10—H101 | 0.960 |
| C1—N7 | 1.313 (4) | C10—H102 | 0.960 |
| C1—S13 | 1.742 (3) | C11—Cl12 | 1.786 (3) |
| N2—C8 | 1.381 (3) | C11—H111 | 0.960 |
| N2—C10 | 1.457 (3) | C11—H112 | 0.960 |
| C3—C4 | 1.376 (4) | S13—C14 | 1.791 (3) |
| C3—C8 | 1.381 (3) | C14—H141 | 0.960 |
| C4—C5 | 1.389 (4) | C14—H142 | 0.960 |

| | | | |
|---|---|---|---|
| C4—N15 | 1.466 (4) | C14—H143 | 0.960 |
| C5—C6 | 1.383 (4) | N15—O16 | 1.207 (3) |
| C6—C9 | 1.388 (4) | N15—O17 | 1.198 (3) |
| N2—C1—N7 | 114.3 (2) | N7—C9—C6 | 129.3 (2) |
| N2—C1—S13 | 120.2 (2) | N2—C10—C11 | 113.3 (3) |
| N7—C1—S13 | 125.4 (2) | N2—C10—H101 | 108.6 |
| C1—N2—C8 | 105.8 (2) | C11—C10—H101 | 108.6 |
| C1—N2—C10 | 128.0 (2) | N2—C10—H102 | 108.4 |
| C8—N2—C10 | 126.1 (2) | C11—C10—H102 | 108.5 |
| H3—C3—C4 | 122.4 | H101—C10—H102 | 109.5 |
| H3—C3—C8 | 122.5 | C10—C11—Cl12 | 112.4 (2) |
| C4—C3—C8 | 115.1 (2) | C10—C11—H111 | 108.6 |
| C3—C4—C5 | 124.1 (3) | Cl12—C11—H111 | 108.7 |
| C3—C4—N15 | 117.6 (2) | C10—C11—H112 | 108.8 |
| C5—C4—N15 | 118.3 (3) | Cl12—C11—H112 | 108.9 |
| C4—C5—H5 | 120.0 | H111—C11—H112 | 109.5 |
| C4—C5—C6 | 120.1 (3) | C1—S13—C14 | 100.00 (16) |
| H5—C5—C6 | 119.9 | S13—C14—H141 | 109.6 |
| C5—C6—H6 | 121.2 | S13—C14—H142 | 109.5 |
| C5—C6—C9 | 117.6 (3) | H141—C14—H142 | 109.5 |
| H6—C6—C9 | 121.2 | S13—C14—H143 | 109.3 |
| C1—N7—C9 | 104.1 (2) | H141—C14—H143 | 109.5 |
| C3—C8—N2 | 131.8 (2) | H142—C14—H143 | 109.5 |
| C3—C8—C9 | 122.6 (3) | C4—N15—O16 | 119.2 (3) |
| N2—C8—C9 | 105.6 (2) | C4—N15—O17 | 119.1 (3) |
| C8—C9—N7 | 110.2 (2) | O16—N15—O17 | 121.7 (3) |
| C8—C9—C6 | 120.5 (2) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C3—H3···O16$^{i}$ | 0.93 | 2.41 | 3.326 (4) | 168 |
| C6—H6···N7$^{ii}$ | 0.93 | 2.57 | 3.499 (4) | 175 |
| C11—H112···O17$^{iii}$ | 0.96 | 2.55 | 3.437 (4) | 154 |
| C10—H102···S13 | 0.96 | 2.68 | 3.168 (3) | 112 |

Symmetry codes: (i) −*x*+2, −*y*+2, −*z*+1; (ii) −*x*+1, −*y*+2, −*z*; (iii) −*x*+1, −*y*+2, −*z*+1.

**Fig. 1**

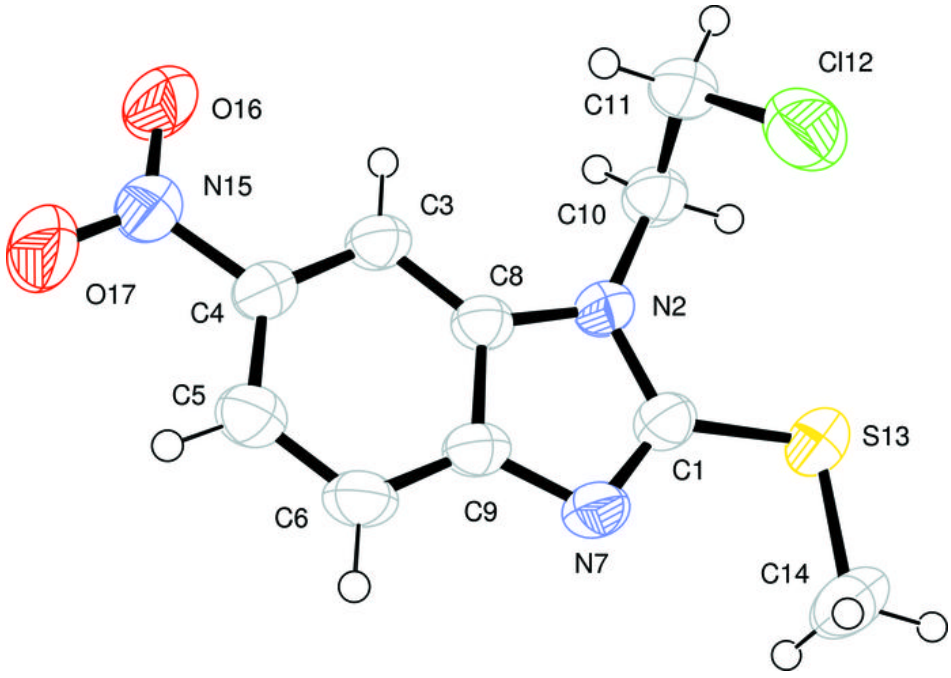

**Fig. 2**