# organic compounds

# 2,5-Dimethyl-3-(3-methylthiophen-2-yl)-perhydropyrrolo[3,4-*d*]isoxazole-4,6-dione

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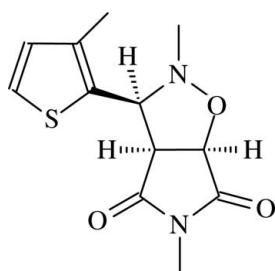Correspondence e-mail: orhanb@omu.edu.tr

The crystal structure of the title compound, $C_{12}H_{14}N_2O_3S$, exhibits intramolecular C—H···S and intermolecular C—H···S, C—H···O hydrogen bonds, C—S···N [S···N = 2.980 (2) Å, C—S···N = 145.78 (17)°] and C—H···$\pi$ interactions; these interactions generate two $C(5)$ chains and $S(4)$, $S(6)$ and $R_4^4(28)$ ring motifs. The isoxazole ring has an envelope conformation; the N atom, which is the flap atom, is displaced by 0.261 (2) Å from the plane defined by the remaining four atoms. The dihedral angle between the succinimide and thiophene rings is 46.8 (2)°.

## Related literature

For general background, see: Huisgen (1960); Black *et al.* (1975); Alibes *et al.* (2003); Kumar *et al.* (2003); Richman (2001); Chiacchio *et al.* (2003*a*,*b*). For ring motif details, see: Etter (1990); Bernstein *et al.* (1995). For related literature, see: Malamidou-Xenikaki *et al.* (1997); Coutouli-Argyropoulou *et al.* (1997); De Clercq (2002*a*,*b*,*c*).



## Experimental

### Crystal data

| | |
|---|---|
| $C_{12}H_{14}N_2O_3S$ | $V$ = 1282.57 (15) Å$^3$ |
| $M_r$ = 266.31 | $Z$ = 4 |
| Orthorhombic, $Pna2_1$ | Mo $K\alpha$ radiation |
| $a$ = 12.0318 (10) Å | $\mu$ = 0.25 mm$^{-1}$ |
| $b$ = 14.6759 (9) Å | $T$ = 296 (2) K |
| $c$ = 7.2635 (4) Å | $0.52 \times 0.48 \times 0.43$ mm |

### Data collection

| | |
|---|---|
| STOE IPDS2 diffractometer | 12725 measured reflections |
| Absorption correction: integration | 2511 independent reflections |
| ($X$-$RED32$; Stoe & Cie, 2002) | 2212 reflections with $I > 2\sigma(I)$ |
| $T_{min}$ = 0.895, $T_{max}$ = 0.929 | $R_{int}$ = 0.058 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.040 | H atoms treated by a mixture of |
| $wR(F^2)$ = 0.090 | independent and constrained |
| $S$ = 1.08 | refinement |
| 2511 reflections | $\Delta\rho_{max}$ = 0.18 e Å$^{-3}$ |
| 177 parameters | $\Delta\rho_{min}$ = −0.16 e Å$^{-3}$ |
| 1 restraint | Absolute structure: Flack (1983), |
| | 1151 Friedel pairs |
| | Flack parameter: 0.01 (9) |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C7—H7C···S1 | 0.96 | 2.97 | 3.467 (3) | 114 |
| C5—H5C···S1$^i$ | 0.96 | 2.91 | 3.808 (3) | 156 |
| C8—H8···O3$^{ii}$ | 0.88 (2) | 2.56 (2) | 3.426 (3) | 168 (2) |
| C12—H12C···Cg1$^{iii}$ | 0.96 | 2.94 | 3.693 (3) | 137 |

Symmetry codes: (i) $x, y, z - 1$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, z$; (iii) $-x + \frac{1}{2}, y + \frac{1}{2}, z + \frac{1}{2}$. Cg1 is the centroid of the S1,C1–C4 ring.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2041).

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# supporting information

## 2,5-Dimethyl-3-(3-methylthiophen-2-yl)perhydropyrrolo[3,4-*d*]isoxazole-4,6-dione

**Mustafa Odabaşoğlu, Hamdi Özkan, Yılmaz Yıldırır and Orhan Büyükgüngör**

### S1. Comment

A general principle for the synthesis of five-membered rings was introduced in 1960 as 1,3-dipolar cycloaddition and turned out to be remarkably widespread (Huisgen, 1960). Because of easy 1,3-dipolar cycloaddition reactions to alkenes, alkynes, isocyanates, isothiocyanates, phospharanes, sulphenes and sulphynl compounds, nitrones are the important intermediates in synthetic organic chemistry (Black *et al.*, 1975). Especially, the nitrone-olefin 1,3-dipolar cycloaddition reaction is interesting since it can create three new contiguous stereogenic centers in a single step (Alibes *et al.*, 2003). Both inter and intramolecular nitrone cycloaddition reactions have received attention because they are useful methods for the formation of heterocycles of biological active compounds (Kumar *et al.*, 2003).

There has been an ever-increasing quest for modified nucleosides due to their potential applications in antiviral and anticancer therapies (Richman, 2001; De Clercq, 2002*a*,b,c). In a recent approach to modified nucleosides, the furanose ring has been replaced by other heterocyclic analogs (Chiacchio *et al.*, 2003*a*). Among these N and O containing five-membered heterocycles, isoxazolidines, and isoxazoline derivatives have emerged as important candidates, and have been shown to display useful anticancer and antiviral properties (Chiacchio *et al.*, 2003*b*). Consequently, synthetic studies on isoxazolidines have drawn considerable attention and 1,3-dipolar cycloadditions of nitrones afford the most straightforward route to isoxazolidines and we have described, the syntheses and crystal structure of, (I), 2,5-di-methyl-3-(3-methylthiophen-2-yl)-dihydro-2*H*-pyrrolo [3,4-*d*]isoxazole-4,6(5*H*,6a*H*)-dione.

The overall view and atom-labeling of the molecule of (I) are displayed in Figure 1. The hydrogen-bonding parameters are given in Table 1 and the packing arrangement of the molecules is illustrated in Figures 2–5. Compound is stabilized by intramolecular C—H···S hydrogen bond and S···N heteroatom interactions [in C1—S1···N; S···N = 2.980 (2) Å, C1—S1···N = 145.78 (17) °], which form S(4) and S(6) motifs, and intermolecular C—H···S and C—H···O hydrogen bonds and C—H···$\pi$ interactions. As shown in Figures 2 and 3 the structure of the compound is made up of C8—H8···O3 and C5—H5c···S1 H-bonded polymeric bands of [$C_{12}H_{14}N_2O_3S$] molecules which are lined up nearly along the diagonal of the (100) (Fig. 2) and (001) (Fig. 3) planes. These polymeric C(5) chains are linked to each other and generate $R_4^4(28)$ ring motifs (Bernstein *et al.*, 1995; Etter, 1990) (Fig. 4). The crystal packing is also stabilized by C12—H12c···Cg1 interactions (Fig. 5, Table 1). The dihedral angle between the succinimide and thiophen rings in [$C_{12} H_{14} N_2 O_3 S$] molecules is 46.8 (2) °.

### S2. Experimental

*N*-Methyl-*C*-(-3-methylthiophen)nitrone was prepared from 3-methylthiophenecarbaldehyde, *N*-methyl-hydroxylamine hydrochloride and sodium carbonate in ethanol according to the procedure previously described (Malamidou-Xenikaki *et al.*, 1997). This substance (3 mmol, 0.465 g) and *N*-methylmaleimide (3.3 mmol, 0.370 g) was dissolved in 50 ml benzene. The reaction mixture was refluxed for 9 h monitored by TLC. After evaporation of the solvent, the reaction

mixture was separated by column chromatography, using mixtures of petroleum ether and ethyl acetate (1:1) as the eluant. The *cis*-isomer, (I), was recrystallized from CHCl₃ / n-hexane (Fig. 6) (mp: 403–405 K).

## S3. Refinement

The aromatic and methyl H atoms included in calculated positions and refined using a riding model approximation with the constrains 0.93–0.96 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic and $U_{iso}(H) = 1.0U_{eq}(C)$ for methyl. The methine H atoms were found in difference Fourier map and refined freely.
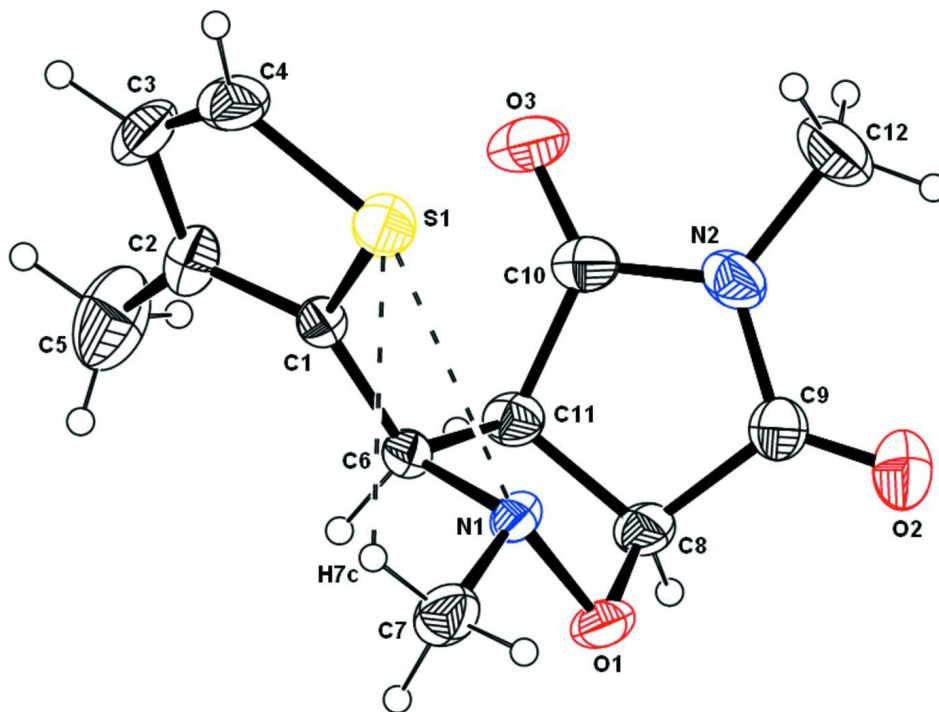


**Figure 1**

A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.
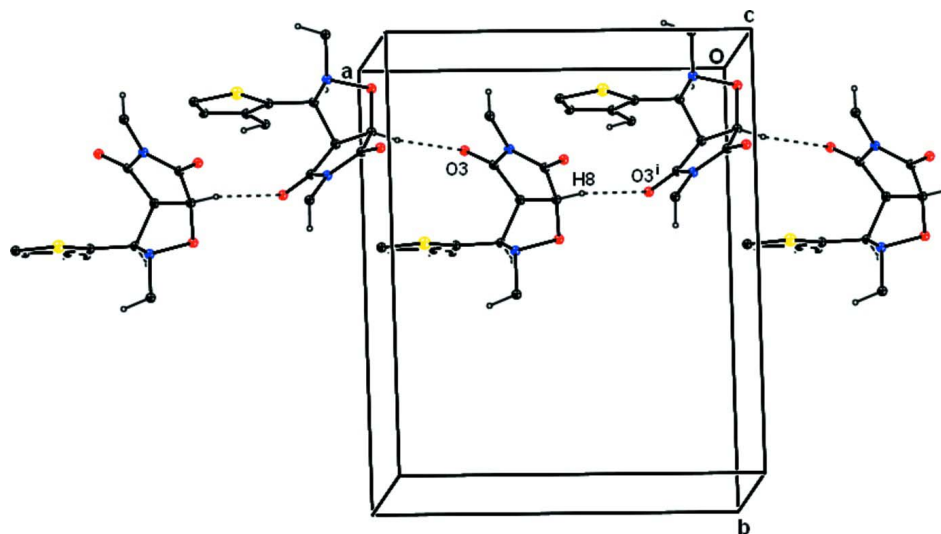
**Figure 2**

Part of the crystal structure of (I), showing the formation of C(5) chain along the [100] direction. H atoms not involved in hydrogen bonds have been omitted for clarity. The dashed line indicates a hydrogen bond. [Symmetry code: (i) 1 - *x*, 0.5 - *y*, *z*].
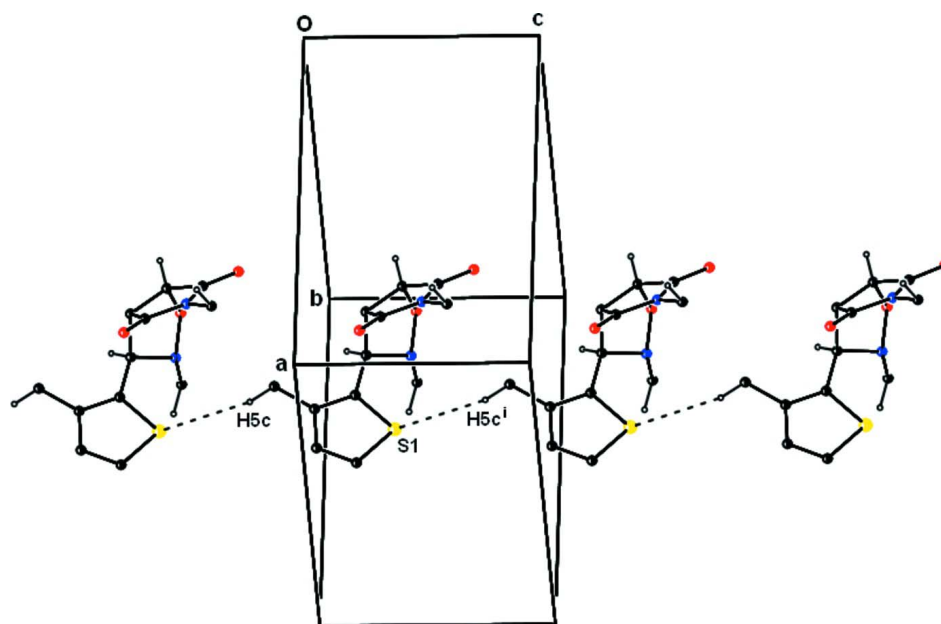


**Figure 3**

Part of the crystal structure of (I), showing the formation of C(5) chain along the [001] direction. H atoms not involved in hydrogen bonds have been omitted for clarity. The dashed line indicates a hydrogen bond. [Symmetry code: (i) *x*, *y*, *z* + 1].
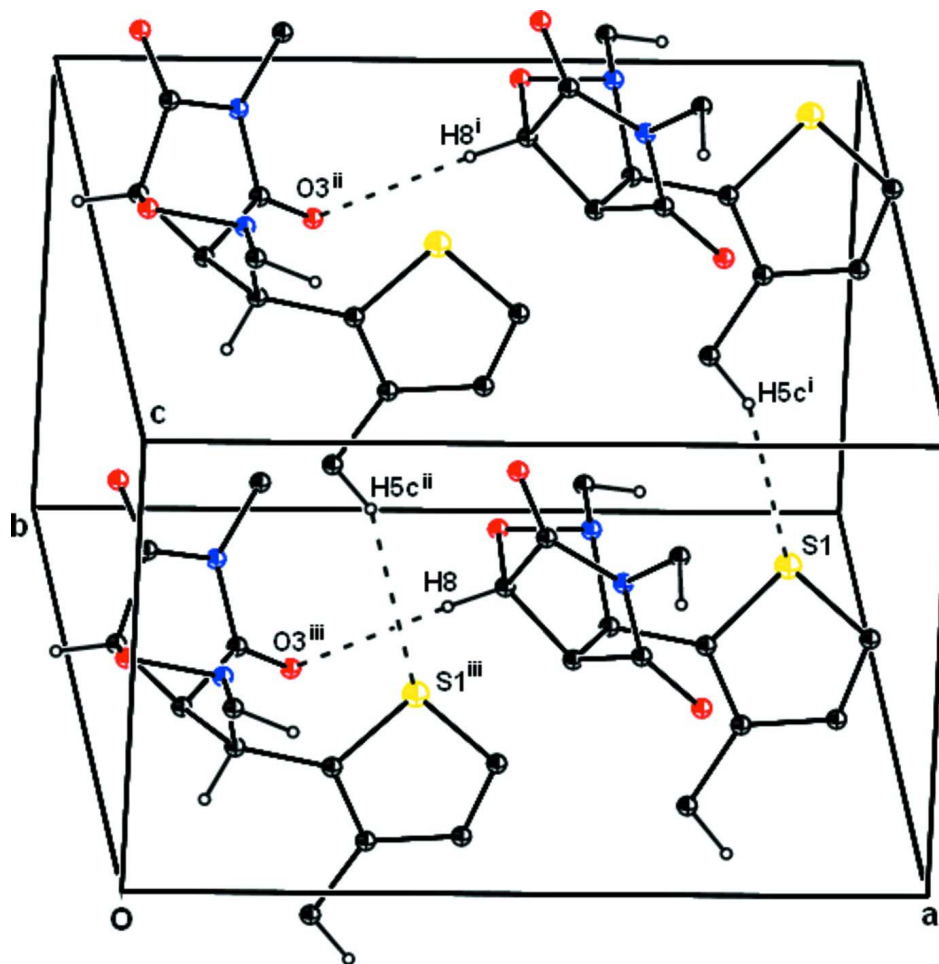
**Figure 4**

Part of the crystal structure of (I), showing the formation of $R_4^4(28)$ ring motif. H atoms not involved in hydrogen bonds have been omitted for clarity. The dashed line indicates hydrogen bond. [Symmetry codes: (i) $x$, $y$, $z + 1$; (ii) $1 - x$, $1/2 - y$, $1 + z$; (iii) $1 - x$, $1/2 - y$, $z$].

**Figure 5**

A packing diagram of (I), with hydrogen bonds drawn as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) 3/2 + $x$, 1/2 + $y$, 1/2 + $z$; (ii) 1 - $x$, 1 - $y$, -$z$; (iii) 1 - $x$, 1 - $y$, $z$ - 1/2; (iv) 1/2 + $x$, 1/2 + $y$, $z$]
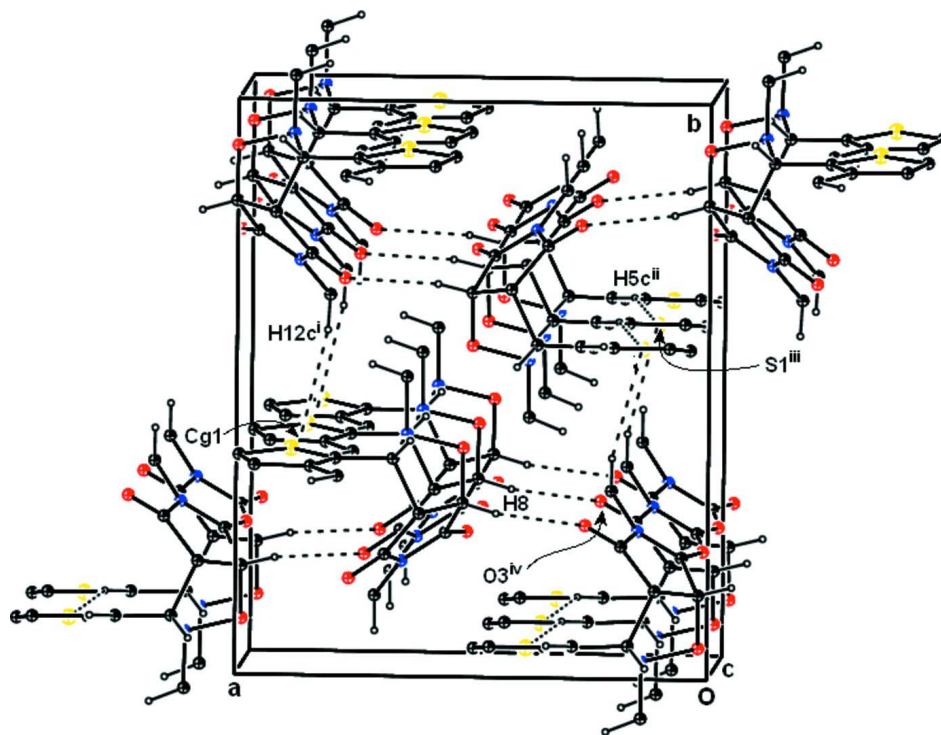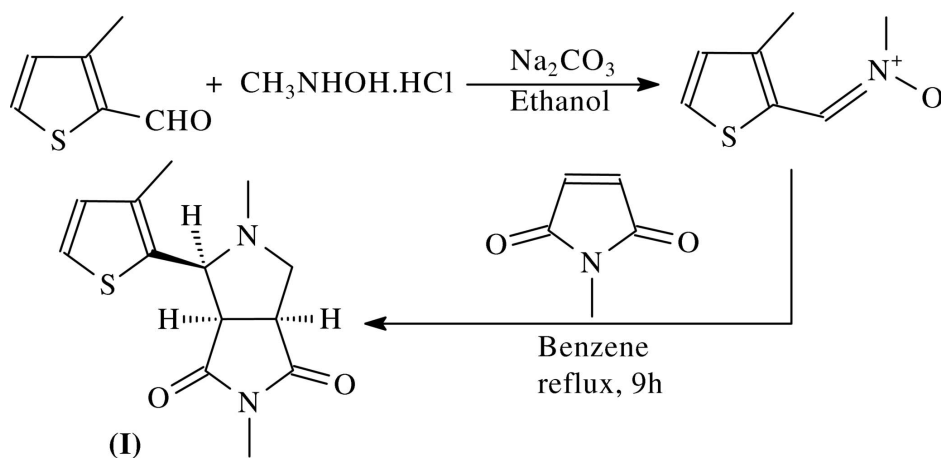


**Figure 6**

Preparation of the title compound.

**2,5-Dimethyl-3-(3-methylthiophen-2-yl)perhydropyrrolo[3,4-*d*]isoxazole- 4,6-dione**

*Crystal data*

| | |
|---|---|
| $C_{12}H_{14}N_2O_3S$ | Hall symbol: P 2c -2n |
| $M_r = 266.31$ | $a = 12.0318$ (10) Å |
| Orthorhombic, $Pna2_1$ | $b = 14.6759$ (9) Å |

$c = 7.2635$ (4) Å

$V = 1282.57$ (15) Å³

$Z = 4$

$F(000) = 560$

$D_x = 1.379$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 12725 reflections

$\theta = 1.7–28.0°$

$\mu = 0.25$ mm⁻¹

$T = 296$ K

Block, colorless

$0.52 \times 0.48 \times 0.43$ mm

*Data collection*

STOE IPDS2
  diffractometer

Radiation source: sealed X-ray tube, 12 x 0.4
  mm long-fine focus

Plane graphite monochromator

Detector resolution: 6.67 pixels mm⁻¹

w–scan rotation method

Absorption correction: integration
  (*X-RED32*; Stoe & Cie, 2002)

$T_{min} = 0.895$, $T_{max} = 0.929$

12725 measured reflections

2511 independent reflections

2212 reflections with $I > 2\sigma(I)$

$R_{int} = 0.058$

$\theta_{max} = 26.0°$, $\theta_{min} = 2.2°$

$h = -14 \rightarrow 14$

$k = -18 \rightarrow 18$

$l = -8 \rightarrow 8$

*Refinement*

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.090$

$S = 1.08$

2511 reflections

177 parameters

1 restraint

Primary atom site location: structure-invariant
  direct methods

Secondary atom site location: difference Fourier
  map

Hydrogen site location: inferred from
  neighbouring sites

H atoms treated by a mixture of independent
  and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.1351P]$
  where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.19$ e Å⁻³

$\Delta\rho_{min} = -0.16$ e Å⁻³

Absolute structure: Flack (1983), 1151 Friedel
  pairs

Absolute structure parameter: 0.01 (9)

*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**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
| --- | --- | --- | --- | --- |
| C1 | 0.76496 (19) | 0.41201 (15) | 0.2036 (3) | 0.0359 (5) |
| C2 | 0.8126 (2) | 0.40438 (19) | 0.0347 (4) | 0.0543 (7) |
| C3 | 0.9305 (2) | 0.4061 (2) | 0.0477 (5) | 0.0651 (9) |
| H3 | 0.9768 | 0.4008 | −0.0543 | 0.078* |
| C4 | 0.9688 (2) | 0.41604 (19) | 0.2179 (5) | 0.0622 (8) |
| H4 | 1.0438 | 0.4198 | 0.2479 | 0.075* |
| C5 | 0.7496 (4) | 0.3958 (3) | −0.1384 (4) | 0.1026 (14) |
| H5A | 0.6863 | 0.4356 | −0.1349 | 0.103* |

| | | | |
|---|---|---|---|
| H5B | 0.7249 | 0.3340 | −0.1529 | 0.103* |
| H5C | 0.7964 | 0.4122 | −0.2401 | 0.103* |
| C6 | 0.64389 (18) | 0.41050 (16) | 0.2458 (3) | 0.0369 (5) |
| C7 | 0.6216 (2) | 0.54462 (18) | 0.4463 (4) | 0.0608 (7) |
| H7A | 0.6049 | 0.5621 | 0.5707 | 0.061* |
| H7B | 0.5670 | 0.5701 | 0.3648 | 0.061* |
| H7C | 0.6939 | 0.5668 | 0.4133 | 0.061* |
| C8 | 0.5012 (2) | 0.32778 (16) | 0.4027 (4) | 0.0477 (6) |
| C9 | 0.5415 (2) | 0.26963 (18) | 0.5620 (4) | 0.0523 (6) |
| C10 | 0.6628 (2) | 0.24093 (17) | 0.3249 (4) | 0.0482 (7) |
| C11 | 0.5881 (2) | 0.31489 (17) | 0.2531 (4) | 0.0441 (5) |
| C12 | 0.6924 (3) | 0.1569 (2) | 0.6175 (6) | 0.0829 (10) |
| H12A | 0.6583 | 0.1543 | 0.7369 | 0.083* |
| H12B | 0.7685 | 0.1756 | 0.6301 | 0.083* |
| H12C | 0.6895 | 0.0978 | 0.5611 | 0.083* |
| N1 | 0.61980 (15) | 0.44503 (14) | 0.4315 (3) | 0.0409 (5) |
| N2 | 0.63330 (18) | 0.22216 (14) | 0.5031 (4) | 0.0546 (6) |
| O1 | 0.50134 (14) | 0.42160 (11) | 0.4512 (3) | 0.0543 (5) |
| O2 | 0.5015 (2) | 0.26507 (16) | 0.7130 (3) | 0.0813 (7) |
| O3 | 0.73562 (16) | 0.20182 (14) | 0.2435 (4) | 0.0697 (6) |
| S1 | 0.86334 (5) | 0.42104 (5) | 0.37501 (8) | 0.05480 (19) |
| H6 | 0.6036 (19) | 0.4459 (14) | 0.160 (3) | 0.028 (5)* |
| H8 | 0.432 (2) | 0.3135 (15) | 0.374 (4) | 0.045 (6)* |
| H11 | 0.561 (2) | 0.2974 (15) | 0.130 (4) | 0.037 (6)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0367 (12) | 0.0340 (11) | 0.0370 (11) | −0.0006 (9) | 0.0001 (9) | 0.0022 (9) |
| C2 | 0.0608 (16) | 0.0580 (16) | 0.0442 (15) | −0.0081 (13) | 0.0116 (13) | −0.0019 (12) |
| C3 | 0.0514 (16) | 0.0625 (18) | 0.082 (2) | −0.0035 (14) | 0.0339 (17) | −0.0026 (16) |
| C4 | 0.0357 (14) | 0.0555 (17) | 0.096 (2) | 0.0034 (12) | 0.0073 (14) | 0.0085 (17) |
| C5 | 0.109 (3) | 0.151 (4) | 0.0478 (17) | −0.020 (3) | 0.011 (2) | −0.020 (2) |
| C6 | 0.0326 (11) | 0.0403 (12) | 0.0378 (11) | 0.0036 (10) | −0.0012 (10) | 0.0071 (10) |
| C7 | 0.0629 (17) | 0.0447 (14) | 0.0747 (18) | 0.0046 (13) | 0.0221 (14) | −0.0023 (13) |
| C8 | 0.0284 (11) | 0.0511 (15) | 0.0634 (18) | −0.0013 (10) | 0.0011 (12) | 0.0058 (12) |
| C9 | 0.0459 (13) | 0.0448 (14) | 0.0664 (17) | −0.0075 (12) | 0.0057 (13) | 0.0077 (12) |
| C10 | 0.0356 (12) | 0.0355 (13) | 0.0735 (18) | −0.0046 (10) | −0.0001 (11) | −0.0017 (11) |
| C11 | 0.0344 (11) | 0.0462 (14) | 0.0517 (15) | −0.0007 (10) | −0.0063 (11) | −0.0006 (11) |
| C12 | 0.082 (2) | 0.061 (2) | 0.106 (3) | 0.0089 (17) | −0.010 (2) | 0.036 (2) |
| N1 | 0.0343 (10) | 0.0412 (10) | 0.0472 (11) | 0.0010 (8) | 0.0110 (8) | 0.0021 (8) |
| N2 | 0.0481 (13) | 0.0400 (11) | 0.0758 (16) | −0.0013 (9) | −0.0049 (11) | 0.0138 (11) |
| O1 | 0.0368 (9) | 0.0461 (9) | 0.0800 (12) | 0.0083 (7) | 0.0194 (8) | 0.0056 (9) |
| O2 | 0.0908 (17) | 0.0780 (15) | 0.0750 (14) | 0.0003 (13) | 0.0242 (13) | 0.0235 (13) |
| O3 | 0.0463 (11) | 0.0520 (11) | 0.1108 (16) | 0.0067 (9) | 0.0152 (12) | −0.0069 (11) |
| S1 | 0.0411 (3) | 0.0754 (4) | 0.0479 (3) | −0.0001 (3) | −0.0090 (3) | 0.0068 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.358 (3) | C7—H7B | 0.9600 |
| C1—C6 | 1.489 (3) | C7—H7C | 0.9600 |
| C1—S1 | 1.723 (2) | C8—O1 | 1.421 (3) |
| C2—C3 | 1.422 (4) | C8—C9 | 1.517 (4) |
| C2—C5 | 1.474 (4) | C8—C11 | 1.519 (4) |
| C3—C4 | 1.328 (5) | C8—H8 | 0.88 (3) |
| C3—H3 | 0.9300 | C9—O2 | 1.200 (3) |
| C4—S1 | 1.708 (3) | C9—N2 | 1.374 (4) |
| C4—H4 | 0.9300 | C10—O3 | 1.202 (3) |
| C5—H5A | 0.9600 | C10—N2 | 1.371 (4) |
| C5—H5B | 0.9600 | C10—C11 | 1.503 (4) |
| C5—H5C | 0.9600 | C11—H11 | 0.99 (3) |
| C6—N1 | 1.470 (3) | C12—N2 | 1.454 (4) |
| C6—C11 | 1.556 (3) | C12—H12A | 0.9600 |
| C6—H6 | 0.95 (2) | C12—H12B | 0.9600 |
| C7—N1 | 1.466 (3) | C12—H12C | 0.9600 |
| C7—H7A | 0.9600 | N1—O1 | 1.473 (2) |
| | | | |
| C2—C1—C6 | 126.7 (2) | O1—C8—C11 | 107.29 (19) |
| C2—C1—S1 | 111.65 (19) | C9—C8—C11 | 104.8 (2) |
| C6—C1—S1 | 121.65 (16) | O1—C8—H8 | 106.9 (15) |
| C1—C2—C3 | 111.1 (2) | C9—C8—H8 | 110.5 (17) |
| C1—C2—C5 | 124.1 (3) | C11—C8—H8 | 116.6 (19) |
| C3—C2—C5 | 124.8 (3) | O2—C9—N2 | 125.4 (3) |
| C4—C3—C2 | 114.2 (3) | O2—C9—C8 | 126.9 (3) |
| C4—C3—H3 | 122.9 | N2—C9—C8 | 107.8 (2) |
| C2—C3—H3 | 122.9 | O3—C10—N2 | 123.9 (3) |
| C3—C4—S1 | 111.6 (2) | O3—C10—C11 | 127.6 (3) |
| C3—C4—H4 | 124.2 | N2—C10—C11 | 108.5 (2) |
| S1—C4—H4 | 124.2 | C10—C11—C8 | 104.7 (2) |
| C2—C5—H5A | 109.5 | C10—C11—C6 | 113.9 (2) |
| C2—C5—H5B | 109.5 | C8—C11—C6 | 102.1 (2) |
| H5A—C5—H5B | 109.5 | C10—C11—H11 | 108.9 (14) |
| C2—C5—H5C | 109.5 | C8—C11—H11 | 117.1 (15) |
| H5A—C5—H5C | 109.5 | C6—C11—H11 | 110.3 (14) |
| H5B—C5—H5C | 109.5 | N2—C12—H12A | 109.5 |
| N1—C6—C1 | 112.12 (19) | N2—C12—H12B | 109.5 |
| N1—C6—C11 | 101.21 (19) | H12A—C12—H12B | 109.5 |
| C1—C6—C11 | 116.27 (19) | N2—C12—H12C | 109.5 |
| N1—C6—H6 | 108.4 (13) | H12A—C12—H12C | 109.5 |
| C1—C6—H6 | 110.9 (14) | H12B—C12—H12C | 109.5 |
| C11—C6—H6 | 107.2 (13) | C7—N1—C6 | 114.11 (19) |
| N1—C7—H7A | 109.5 | C7—N1—O1 | 103.87 (18) |
| N1—C7—H7B | 109.5 | C6—N1—O1 | 101.51 (17) |
| H7A—C7—H7B | 109.5 | C10—N2—C9 | 113.6 (2) |
| N1—C7—H7C | 109.5 | C10—N2—C12 | 123.1 (3) |

| | | | |
|---|---|---|---|
| H7A—C7—H7C | 109.5 | C9—N2—C12 | 123.3 (3) |
| H7B—C7—H7C | 109.5 | C8—O1—N1 | 101.71 (16) |
| O1—C8—C9 | 110.8 (2) | C4—S1—C1 | 91.40 (14) |
| | | | |
| C6—C1—C2—C3 | −178.3 (2) | N1—C6—C11—C10 | −88.1 (2) |
| S1—C1—C2—C3 | −0.1 (3) | C1—C6—C11—C10 | 33.7 (3) |
| C6—C1—C2—C5 | 1.9 (5) | N1—C6—C11—C8 | 24.1 (2) |
| S1—C1—C2—C5 | −179.9 (3) | C1—C6—C11—C8 | 145.9 (2) |
| C1—C2—C3—C4 | −1.0 (4) | C1—C6—N1—C7 | 78.5 (3) |
| C5—C2—C3—C4 | 178.8 (3) | C11—C6—N1—C7 | −156.9 (2) |
| C2—C3—C4—S1 | 1.5 (4) | C1—C6—N1—O1 | −170.41 (17) |
| C2—C1—C6—N1 | −164.3 (2) | C11—C6—N1—O1 | −45.82 (19) |
| S1—C1—C6—N1 | 17.6 (3) | O3—C10—N2—C9 | −174.5 (3) |
| C2—C1—C6—C11 | 79.9 (3) | C11—C10—N2—C9 | 4.2 (3) |
| S1—C1—C6—C11 | −98.2 (2) | O3—C10—N2—C12 | 4.5 (4) |
| O1—C8—C9—O2 | 59.0 (4) | C11—C10—N2—C12 | −176.8 (3) |
| C11—C8—C9—O2 | 174.4 (3) | O2—C9—N2—C10 | −179.0 (3) |
| O1—C8—C9—N2 | −121.1 (2) | C8—C9—N2—C10 | 1.0 (3) |
| C11—C8—C9—N2 | −5.6 (3) | O2—C9—N2—C12 | 2.0 (5) |
| O3—C10—C11—C8 | 171.1 (2) | C8—C9—N2—C12 | −177.9 (3) |
| N2—C10—C11—C8 | −7.5 (3) | C9—C8—O1—N1 | 78.9 (2) |
| O3—C10—C11—C6 | −78.2 (3) | C11—C8—O1—N1 | −34.9 (2) |
| N2—C10—C11—C6 | 103.1 (2) | C7—N1—O1—C8 | 169.8 (2) |
| O1—C8—C11—C10 | 125.6 (2) | C6—N1—O1—C8 | 51.1 (2) |
| C9—C8—C11—C10 | 7.7 (3) | C3—C4—S1—C1 | −1.3 (2) |
| O1—C8—C11—C6 | 6.7 (2) | C2—C1—S1—C4 | 0.8 (2) |
| C9—C8—C11—C6 | −111.2 (2) | C6—C1—S1—C4 | 179.1 (2) |

*Hydrogen-bond geometry (Å, º)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| C7—H7*C*···S1 | 0.96 | 2.97 | 3.467 (3) | 114 |
| C5—H5*C*···S1[i] | 0.96 | 2.91 | 3.808 (3) | 156 |
| C8—H8···O3[ii] | 0.88 (2) | 2.56 (2) | 3.426 (3) | 168 (2) |
| C12—H12*C*···*Cg*1[iii] | 0.96 | 2.94 | 3.693 (3) | 137 |

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