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1-[2-(2,4-Dinitrobenzylideneamino)-phenyl]-3-phenylthiourea

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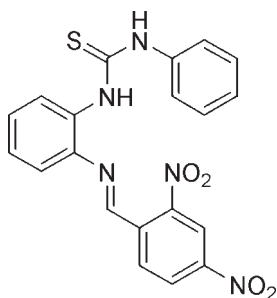
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 Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.046; wR factor = 0.137; data-to-parameter ratio = 25.4.

In the title compound, $\text{C}_{20}\text{H}_{15}\text{N}_5\text{O}_4\text{S}$, the central benzene ring makes dihedral angles of 59.5 (1) and 51.7 (1)°, respectively, with the terminal phenyl and benzene rings. The molecular structure exhibits weak intramolecular $\text{N}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\text{S}$ interactions. In the crystal structure, molecules are linked by weak intermolecular $\text{N}-\text{H}\cdots\text{S}$ and $\text{C}-\text{H}\cdots\text{O}$ interactions, forming a chain along $[\bar{1}\bar{1}1]$.

Related literature

For the biological activity of thioureas, see: Huebner *et al.* (1953); Madan & Taneja (1991); Manjula *et al.* (2009). For related structures, see: Gayathri *et al.* (2007, 2008). For graph-set notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

 $\text{C}_{20}\text{H}_{15}\text{N}_5\text{O}_4\text{S}$
 $M_r = 421.43$

 Monoclinic, $P2_1/c$
 $a = 8.362$ (5) Å

 $b = 18.767$ (3) Å
 $c = 12.379$ (4) Å
 $\beta = 94.827$ (5)°
 $V = 1935.7$ (14) Å³
 $Z = 4$

 Mo $K\alpha$ radiation
 $\mu = 0.21$ mm⁻¹
 $T = 295$ K
 $0.20 \times 0.16 \times 0.16$ mm

Data collection

 Bruker Kappa APEXII
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.960$, $T_{\max} = 0.968$

 28336 measured reflections
 6878 independent reflections
 4509 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.137$
 $S = 1.02$
 6878 reflections

 271 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.25$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.37$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{N4}-\text{H4A}\cdots\text{N3}$ | 0.86 | 2.14 | 2.614 (2) | 114 |
| $\text{C3}-\text{H3}\cdots\text{S1}$ | 0.93 | 2.55 | 3.215 (2) | 128 |
| $\text{N4}-\text{H4A}\cdots\text{N3}$ | 0.86 | 2.14 | 2.614 (2) | 114 |
| $\text{N5}-\text{H5A}\cdots\text{S1}^i$ | 0.86 | 2.49 | 3.284 (2) | 155 |
| $\text{C12}-\text{H12}\cdots\text{O3}^{ii}$ | 0.93 | 2.57 | 3.397 (3) | 148 |

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

The authors wish to acknowledge IIT, Madras for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2457).

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supplementary materials

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1-[2-(2,4-Dinitrobenzylideneamino)phenyl]-3-phenylthiourea

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Comment

Thioureas are known to exhibit antiviral, antibacterial, anticancer (Madan & Taneja, 1991; Manjula *et al.*, 2009), antifungal, antitubercular, antithyroidal, herbicidal and insecticidal (Huebner *et al.*, 1953) activities.

The geometric parameters in (I), (Fig. 1) agree with the reported values of similar structures (Gayathri *et al.*, 2007, 2008). The benzene ring C1—C6 makes the dihedral angle of 59.5 (1)° with the phenyl ring C15—C20 and 51.7 (1)° with the dinitrobenzene ring C8—C13.

The molecular structure of (I) exhibits weak intramolecular N—H⋯N, C—H⋯S and C—H⋯O interactions and the crystal structure is stabilized by weak intermolecular N—H⋯S and C—H⋯O interactions (Table 1 and Fig. 2). The intermolecular N5—H5A⋯S1 interaction generates an eight-membered ring, with graph-set motif $R_2^2(8)$ and the C12—H12⋯O3 interaction generates a ten-membered ring, with graph-set motif $R_2^2(10)$ (Bernstein, 1995).

Experimental

To the solution of 1-(2-aminophenyl)-3-phenylthiourea (0.3 g, 1.2 mmol) in methanol (25 ml), 2,4-dinitrobenzaldehyde (0.36 g, 1.2 mmol) in methanol (25 ml) was added under stirring. The resulting mixture was refluxed for 3 h and cooled to room temperature. The solid product was collected by filtration and washed with cold methanol. The microcrystalline compound was recrystallized from hot chloroform.

Refinement

H atoms were positioned geometrically and refined using riding model, with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, and with N—H = 0.86 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

Figures

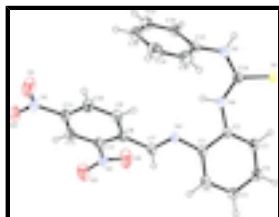


Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

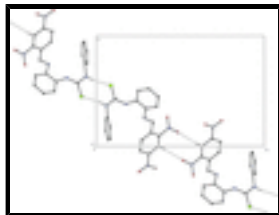


Fig. 2. The packing of (I), viewed down the a axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

1-[2-(2,4-Dinitrobenzylideneamino)phenyl]-3-phenylthiourea

Crystal data

| | |
|---------------------------------|---|
| $C_{20}H_{15}N_5O_4S$ | $F_{000} = 872$ |
| $M_r = 421.43$ | $D_x = 1.446 \text{ Mg m}^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| Hall symbol: -P 2ybc | Cell parameters from 9702 reflections |
| $a = 8.362 (5) \text{ \AA}$ | $\theta = 2.2\text{--}32.3^\circ$ |
| $b = 18.767 (3) \text{ \AA}$ | $\mu = 0.21 \text{ mm}^{-1}$ |
| $c = 12.379 (4) \text{ \AA}$ | $T = 295 \text{ K}$ |
| $\beta = 94.827 (5)^\circ$ | Prism, orange |
| $V = 1935.7 (14) \text{ \AA}^3$ | $0.20 \times 0.16 \times 0.16 \text{ mm}$ |
| $Z = 4$ | |

Data collection

| | |
|---|--|
| Bruker Kappa APEXII diffractometer | 6878 independent reflections |
| Radiation source: fine-focus sealed tube | 4509 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.028$ |
| $T = 295 \text{ K}$ | $\theta_{\text{max}} = 32.3^\circ$ |
| ω and φ scans | $\theta_{\text{min}} = 2.2^\circ$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h = -12 \rightarrow 12$ |
| $T_{\text{min}} = 0.960$, $T_{\text{max}} = 0.968$ | $k = -28 \rightarrow 26$ |
| 28336 measured reflections | $l = -9 \rightarrow 18$ |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.046$ | H-atom parameters constrained |
| $wR(F^2) = 0.137$ | $w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.3472P]$ |
| $S = 1.02$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 6878 reflections | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 271 parameters | $\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$ |
| | $\Delta\rho_{\text{min}} = -0.37 \text{ e \AA}^{-3}$ |

Primary atom site location: structure-invariant direct methods Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|--------------|---------------|----------------------------------|
| C1 | 0.62398 (16) | 0.29698 (7) | 0.34283 (10) | 0.0381 (3) |
| C2 | 0.65955 (15) | 0.23913 (7) | 0.41328 (9) | 0.0360 (3) |
| C3 | 0.76661 (18) | 0.24941 (8) | 0.50420 (11) | 0.0448 (3) |
| H3 | 0.7860 | 0.2127 | 0.5541 | 0.054* |
| C4 | 0.84381 (19) | 0.31360 (9) | 0.52044 (12) | 0.0511 (4) |
| H4 | 0.9149 | 0.3200 | 0.5816 | 0.061* |
| C5 | 0.8174 (2) | 0.36867 (9) | 0.44751 (13) | 0.0560 (4) |
| H5 | 0.8740 | 0.4111 | 0.4576 | 0.067* |
| C6 | 0.70627 (19) | 0.36049 (8) | 0.35919 (12) | 0.0496 (4) |
| H6 | 0.6868 | 0.3979 | 0.3106 | 0.060* |
| C7 | 0.42126 (16) | 0.33645 (8) | 0.21914 (10) | 0.0412 (3) |
| H7 | 0.4321 | 0.3809 | 0.2522 | 0.049* |
| C8 | 0.30541 (16) | 0.32650 (7) | 0.12401 (10) | 0.0381 (3) |
| C9 | 0.32496 (18) | 0.26958 (8) | 0.05491 (11) | 0.0459 (3) |
| H9 | 0.4040 | 0.2359 | 0.0739 | 0.055* |
| C10 | 0.23061 (19) | 0.26145 (9) | -0.04113 (11) | 0.0478 (3) |
| H10 | 0.2441 | 0.2225 | -0.0859 | 0.057* |
| C11 | 0.11616 (16) | 0.31229 (8) | -0.06909 (10) | 0.0425 (3) |
| C12 | 0.08777 (17) | 0.36881 (8) | -0.00335 (11) | 0.0424 (3) |
| H12 | 0.0087 | 0.4023 | -0.0234 | 0.051* |
| C13 | 0.18123 (16) | 0.37421 (8) | 0.09399 (10) | 0.0393 (3) |
| C14 | 0.57689 (16) | 0.11081 (7) | 0.42739 (10) | 0.0387 (3) |
| C15 | 0.41735 (19) | 0.06746 (7) | 0.26115 (11) | 0.0435 (3) |
| C16 | 0.2548 (2) | 0.05512 (8) | 0.24467 (12) | 0.0471 (3) |
| H16 | 0.1955 | 0.0457 | 0.3034 | 0.057* |
| C17 | 0.1798 (2) | 0.05672 (10) | 0.14094 (14) | 0.0606 (4) |
| H17 | 0.0700 | 0.0482 | 0.1298 | 0.073* |
| C18 | 0.2668 (3) | 0.07077 (10) | 0.05446 (13) | 0.0687 (5) |
| H18 | 0.2161 | 0.0721 | -0.0153 | 0.082* |
| C19 | 0.4278 (3) | 0.08286 (11) | 0.07051 (14) | 0.0703 (5) |
| H19 | 0.4862 | 0.0923 | 0.0114 | 0.084* |
| C20 | 0.5058 (2) | 0.08133 (10) | 0.17423 (13) | 0.0599 (4) |
| H20 | 0.6157 | 0.0895 | 0.1849 | 0.072* |
| N1 | 0.01932 (16) | 0.30558 (9) | -0.17349 (10) | 0.0534 (3) |
| N2 | 0.14010 (17) | 0.43172 (8) | 0.16707 (11) | 0.0542 (3) |
| N3 | 0.50634 (14) | 0.28461 (6) | 0.25608 (9) | 0.0406 (3) |
| N4 | 0.58017 (14) | 0.17574 (6) | 0.38222 (8) | 0.0409 (3) |
| H4A | 0.5216 | 0.1791 | 0.3219 | 0.049* |
| N5 | 0.49272 (17) | 0.06127 (7) | 0.36821 (10) | 0.0529 (3) |
| H5A | 0.4832 | 0.0206 | 0.3990 | 0.064* |
| O1 | 0.0455 (2) | 0.25501 (10) | -0.23077 (11) | 0.0917 (5) |
| O2 | -0.08088 (15) | 0.35115 (8) | -0.19739 (9) | 0.0652 (3) |
| O3 | 0.0704 (2) | 0.48301 (8) | 0.12851 (13) | 0.0930 (5) |

supplementary materials

| | | | | |
|----|-------------|-------------|--------------|--------------|
| O4 | 0.1705 (2) | 0.42321 (9) | 0.26429 (11) | 0.0902 (5) |
| S1 | 0.66368 (5) | 0.08789 (2) | 0.55035 (3) | 0.04838 (12) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|--------------|---------------|---------------|--------------|
| C1 | 0.0389 (7) | 0.0402 (7) | 0.0341 (5) | 0.0022 (5) | -0.0038 (5) | 0.0060 (5) |
| C2 | 0.0366 (7) | 0.0377 (7) | 0.0330 (5) | 0.0040 (5) | -0.0017 (5) | 0.0047 (5) |
| C3 | 0.0487 (8) | 0.0449 (8) | 0.0384 (6) | 0.0049 (6) | -0.0100 (5) | 0.0062 (5) |
| C4 | 0.0529 (9) | 0.0506 (9) | 0.0465 (7) | 0.0005 (7) | -0.0159 (6) | -0.0013 (6) |
| C5 | 0.0595 (10) | 0.0439 (8) | 0.0609 (9) | -0.0082 (7) | -0.0171 (7) | 0.0021 (7) |
| C6 | 0.0544 (9) | 0.0403 (8) | 0.0516 (8) | -0.0045 (6) | -0.0108 (6) | 0.0107 (6) |
| C7 | 0.0429 (7) | 0.0426 (7) | 0.0368 (6) | 0.0018 (6) | -0.0041 (5) | 0.0065 (5) |
| C8 | 0.0371 (7) | 0.0428 (7) | 0.0335 (5) | 0.0017 (5) | -0.0012 (5) | 0.0091 (5) |
| C9 | 0.0442 (8) | 0.0509 (8) | 0.0416 (6) | 0.0108 (6) | -0.0018 (5) | 0.0056 (6) |
| C10 | 0.0498 (8) | 0.0550 (9) | 0.0381 (6) | 0.0062 (7) | 0.0015 (6) | -0.0022 (6) |
| C11 | 0.0374 (7) | 0.0572 (9) | 0.0322 (5) | -0.0021 (6) | -0.0015 (5) | 0.0077 (5) |
| C12 | 0.0375 (7) | 0.0482 (8) | 0.0405 (6) | 0.0042 (6) | -0.0030 (5) | 0.0106 (5) |
| C13 | 0.0403 (7) | 0.0408 (7) | 0.0364 (6) | 0.0024 (6) | 0.0002 (5) | 0.0062 (5) |
| C14 | 0.0401 (7) | 0.0392 (7) | 0.0364 (6) | 0.0017 (5) | -0.0001 (5) | 0.0072 (5) |
| C15 | 0.0586 (9) | 0.0340 (7) | 0.0369 (6) | -0.0010 (6) | -0.0032 (6) | 0.0032 (5) |
| C16 | 0.0558 (9) | 0.0397 (7) | 0.0455 (7) | 0.0040 (7) | 0.0019 (6) | -0.0005 (6) |
| C17 | 0.0665 (11) | 0.0515 (10) | 0.0602 (9) | 0.0048 (8) | -0.0163 (8) | -0.0025 (7) |
| C18 | 0.1052 (16) | 0.0567 (11) | 0.0405 (7) | 0.0006 (10) | -0.0150 (8) | -0.0024 (7) |
| C19 | 0.1034 (17) | 0.0702 (12) | 0.0388 (7) | -0.0056 (11) | 0.0143 (9) | 0.0021 (7) |
| C20 | 0.0657 (11) | 0.0657 (11) | 0.0489 (8) | -0.0085 (9) | 0.0079 (7) | 0.0050 (7) |
| N1 | 0.0498 (7) | 0.0732 (10) | 0.0357 (5) | -0.0006 (7) | -0.0040 (5) | 0.0038 (6) |
| N2 | 0.0549 (8) | 0.0525 (8) | 0.0531 (7) | 0.0126 (6) | -0.0072 (6) | -0.0041 (6) |
| N3 | 0.0413 (6) | 0.0429 (6) | 0.0360 (5) | -0.0013 (5) | -0.0064 (4) | 0.0103 (4) |
| N4 | 0.0464 (6) | 0.0384 (6) | 0.0358 (5) | -0.0013 (5) | -0.0096 (4) | 0.0074 (4) |
| N5 | 0.0704 (9) | 0.0438 (7) | 0.0420 (6) | -0.0131 (6) | -0.0106 (6) | 0.0141 (5) |
| O1 | 0.1080 (12) | 0.1083 (13) | 0.0534 (7) | 0.0283 (10) | -0.0248 (7) | -0.0255 (8) |
| O2 | 0.0583 (7) | 0.0850 (9) | 0.0490 (6) | 0.0074 (6) | -0.0157 (5) | 0.0116 (6) |
| O3 | 0.1231 (13) | 0.0625 (9) | 0.0886 (10) | 0.0423 (9) | -0.0200 (9) | -0.0069 (7) |
| O4 | 0.1129 (13) | 0.1065 (12) | 0.0489 (7) | 0.0467 (10) | -0.0073 (7) | -0.0162 (7) |
| S1 | 0.0547 (2) | 0.0481 (2) | 0.04016 (17) | -0.00134 (16) | -0.00860 (14) | 0.01530 (14) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-------|-------------|---------|-------------|
| C1—C6 | 1.383 (2) | C12—H12 | 0.9300 |
| C1—C2 | 1.4085 (17) | C13—N2 | 1.468 (2) |
| C1—N3 | 1.4132 (16) | C14—N4 | 1.3419 (17) |
| C2—C3 | 1.3914 (18) | C14—N5 | 1.3452 (19) |
| C2—N4 | 1.4003 (17) | C14—S1 | 1.6860 (13) |
| C3—C4 | 1.374 (2) | C15—C16 | 1.377 (2) |
| C3—H3 | 0.9300 | C15—C20 | 1.381 (2) |
| C4—C5 | 1.378 (2) | C15—N5 | 1.4236 (17) |
| C4—H4 | 0.9300 | C16—C17 | 1.381 (2) |
| C5—C6 | 1.383 (2) | C16—H16 | 0.9300 |

| | | | |
|-------------|-------------|-------------|-------------|
| C5—H5 | 0.9300 | C17—C18 | 1.370 (3) |
| C6—H6 | 0.9300 | C17—H17 | 0.9300 |
| C7—N3 | 1.2678 (17) | C18—C19 | 1.363 (3) |
| C7—C8 | 1.4725 (18) | C18—H18 | 0.9300 |
| C7—H7 | 0.9300 | C19—C20 | 1.391 (2) |
| C8—C9 | 1.387 (2) | C19—H19 | 0.9300 |
| C8—C13 | 1.3975 (19) | C20—H20 | 0.9300 |
| C9—C10 | 1.379 (2) | N1—O1 | 1.215 (2) |
| C9—H9 | 0.9300 | N1—O2 | 1.2162 (19) |
| C10—C11 | 1.375 (2) | N2—O3 | 1.2031 (18) |
| C10—H10 | 0.9300 | N2—O4 | 1.2194 (18) |
| C11—C12 | 1.370 (2) | N4—H4A | 0.8600 |
| C11—N1 | 1.4716 (17) | N5—H5A | 0.8600 |
| C12—C13 | 1.3837 (18) | | |
| C6—C1—C2 | 119.82 (12) | C12—C13—N2 | 116.50 (12) |
| C6—C1—N3 | 123.98 (12) | C8—C13—N2 | 120.97 (12) |
| C2—C1—N3 | 116.18 (12) | N4—C14—N5 | 115.39 (11) |
| C3—C2—N4 | 126.71 (12) | N4—C14—S1 | 125.83 (11) |
| C3—C2—C1 | 118.81 (13) | N5—C14—S1 | 118.76 (10) |
| N4—C2—C1 | 114.48 (11) | C16—C15—C20 | 120.20 (14) |
| C4—C3—C2 | 120.16 (13) | C16—C15—N5 | 118.58 (13) |
| C4—C3—H3 | 119.9 | C20—C15—N5 | 121.10 (15) |
| C2—C3—H3 | 119.9 | C15—C16—C17 | 119.97 (15) |
| C3—C4—C5 | 120.97 (13) | C15—C16—H16 | 120.0 |
| C3—C4—H4 | 119.5 | C17—C16—H16 | 120.0 |
| C5—C4—H4 | 119.5 | C18—C17—C16 | 120.07 (18) |
| C4—C5—C6 | 119.67 (15) | C18—C17—H17 | 120.0 |
| C4—C5—H5 | 120.2 | C16—C17—H17 | 120.0 |
| C6—C5—H5 | 120.2 | C19—C18—C17 | 120.09 (15) |
| C5—C6—C1 | 120.30 (13) | C19—C18—H18 | 120.0 |
| C5—C6—H6 | 119.9 | C17—C18—H18 | 120.0 |
| C1—C6—H6 | 119.9 | C18—C19—C20 | 120.80 (17) |
| N3—C7—C8 | 120.17 (13) | C18—C19—H19 | 119.6 |
| N3—C7—H7 | 119.9 | C20—C19—H19 | 119.6 |
| C8—C7—H7 | 119.9 | C15—C20—C19 | 118.87 (18) |
| C9—C8—C13 | 116.94 (12) | C15—C20—H20 | 120.6 |
| C9—C8—C7 | 119.16 (12) | C19—C20—H20 | 120.6 |
| C13—C8—C7 | 123.75 (13) | O1—N1—O2 | 124.14 (13) |
| C10—C9—C8 | 121.91 (13) | O1—N1—C11 | 117.79 (14) |
| C10—C9—H9 | 119.0 | O2—N1—C11 | 118.06 (14) |
| C8—C9—H9 | 119.0 | O3—N2—O4 | 123.37 (15) |
| C11—C10—C9 | 118.42 (14) | O3—N2—C13 | 118.34 (13) |
| C11—C10—H10 | 120.8 | O4—N2—C13 | 118.15 (13) |
| C9—C10—H10 | 120.8 | C7—N3—C1 | 118.79 (12) |
| C12—C11—C10 | 122.65 (12) | C14—N4—C2 | 133.07 (11) |
| C12—C11—N1 | 118.55 (13) | C14—N4—H4A | 113.5 |
| C10—C11—N1 | 118.80 (14) | C2—N4—H4A | 113.5 |
| C11—C12—C13 | 117.43 (13) | C14—N5—C15 | 128.38 (12) |
| C11—C12—H12 | 121.3 | C14—N5—H5A | 115.8 |

supplementary materials

| | | | |
|-----------------|--------------|-----------------|--------------|
| C13—C12—H12 | 121.3 | C15—N5—H5A | 115.8 |
| C12—C13—C8 | 122.50 (13) | | |
| C6—C1—C2—C3 | 6.0 (2) | N5—C15—C16—C17 | -176.32 (14) |
| N3—C1—C2—C3 | -175.84 (12) | C15—C16—C17—C18 | -0.2 (2) |
| C6—C1—C2—N4 | -173.89 (14) | C16—C17—C18—C19 | 0.3 (3) |
| N3—C1—C2—N4 | 4.25 (18) | C17—C18—C19—C20 | -0.2 (3) |
| N4—C2—C3—C4 | 175.60 (15) | C16—C15—C20—C19 | 0.3 (3) |
| C1—C2—C3—C4 | -4.3 (2) | N5—C15—C20—C19 | 176.38 (16) |
| C2—C3—C4—C5 | -0.2 (3) | C18—C19—C20—C15 | -0.1 (3) |
| C3—C4—C5—C6 | 3.0 (3) | C12—C11—N1—O1 | -179.74 (16) |
| C4—C5—C6—C1 | -1.2 (3) | C10—C11—N1—O1 | -0.1 (2) |
| C2—C1—C6—C5 | -3.3 (2) | C12—C11—N1—O2 | 0.8 (2) |
| N3—C1—C6—C5 | 178.69 (15) | C10—C11—N1—O2 | -179.62 (15) |
| N3—C7—C8—C9 | -20.9 (2) | C12—C13—N2—O3 | -24.3 (2) |
| N3—C7—C8—C13 | 163.79 (13) | C8—C13—N2—O3 | 157.70 (17) |
| C13—C8—C9—C10 | 2.3 (2) | C12—C13—N2—O4 | 151.72 (17) |
| C7—C8—C9—C10 | -173.30 (14) | C8—C13—N2—O4 | -26.3 (2) |
| C8—C9—C10—C11 | 1.1 (2) | C8—C7—N3—C1 | 175.78 (12) |
| C9—C10—C11—C12 | -2.8 (2) | C6—C1—N3—C7 | -31.3 (2) |
| C9—C10—C11—N1 | 177.57 (13) | C2—C1—N3—C7 | 150.61 (13) |
| C10—C11—C12—C13 | 0.9 (2) | N5—C14—N4—C2 | -175.70 (14) |
| N1—C11—C12—C13 | -179.52 (12) | S1—C14—N4—C2 | 5.9 (2) |
| C11—C12—C13—C8 | 2.9 (2) | C3—C2—N4—C14 | 2.5 (2) |
| C11—C12—C13—N2 | -175.14 (13) | C1—C2—N4—C14 | -177.57 (14) |
| C9—C8—C13—C12 | -4.4 (2) | N4—C14—N5—C15 | 5.2 (2) |
| C7—C8—C13—C12 | 171.00 (13) | S1—C14—N5—C15 | -176.34 (13) |
| C9—C8—C13—N2 | 173.51 (13) | C16—C15—N5—C14 | -121.98 (17) |
| C7—C8—C13—N2 | -11.1 (2) | C20—C15—N5—C14 | 61.8 (2) |
| C20—C15—C16—C17 | -0.1 (2) | | |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|-------|-------------|-------------|---------------|
| N4—H4A \cdots N3 | 0.86 | 2.14 | 2.614 (2) | 114 |
| C3—H3 \cdots S1 | 0.93 | 2.55 | 3.215 (2) | 128 |
| C7—H7 \cdots O4 | 0.93 | 2.34 | 2.749 (3) | 106 |
| N4—H4A \cdots N3 | 0.86 | 2.14 | 2.614 (2) | 114 |
| N5—H5A \cdots S1 ⁱ | 0.86 | 2.49 | 3.284 (2) | 155 |
| C12—H12 \cdots O3 ⁱⁱ | 0.93 | 2.57 | 3.397 (3) | 148 |

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

Fig. 1

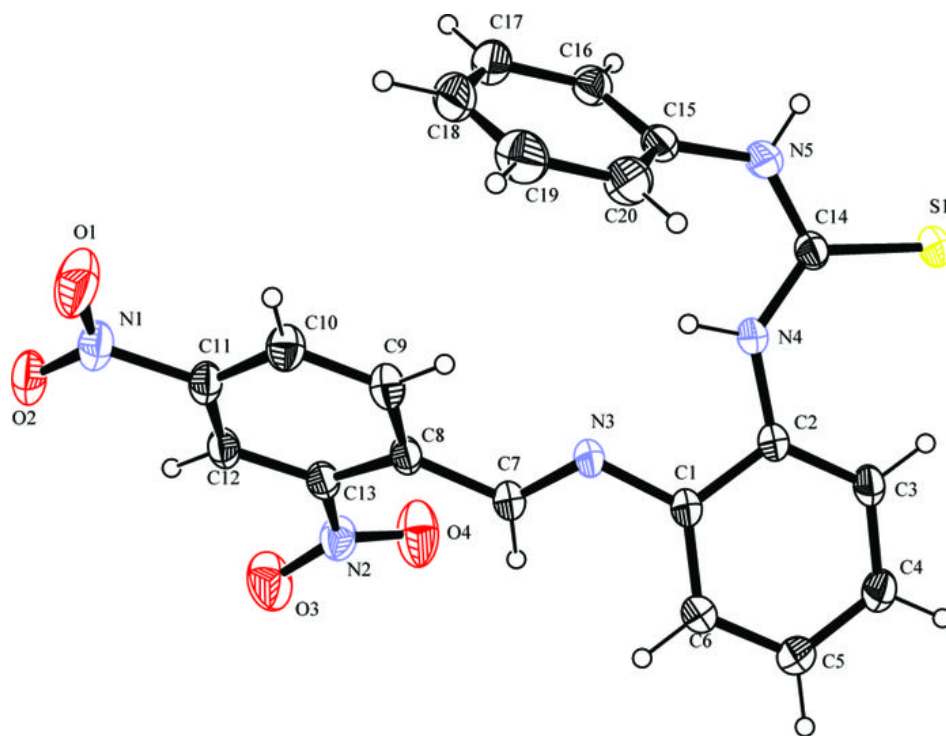


Fig. 2

