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4-(4-Chlorophenyl)-1-(2-hydroxy-2,2-diphenylacetyl)thiosemicarbazide

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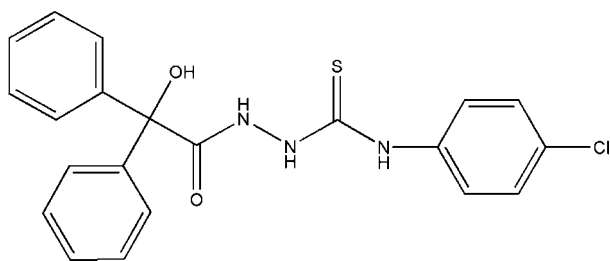
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 Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.041; wR factor = 0.102; data-to-parameter ratio = 17.0.

The asymmetric unit of the title compound, $\text{C}_{21}\text{H}_{18}\text{ClN}_3\text{O}_2\text{S}$, contains two molecules in which the bond lengths and angles are almost identical. Intramolecular $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds result in the formation of two five-membered rings. In the crystal structure, intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into centrosymmetric dimers; these dimers are linked *via* intermolecular $\text{O}-\text{H}\cdots\text{S}$ hydrogen bonds, leading to infinite corrugated layers parallel to the bc plane through $R_2^2(16)$ ring motifs.

Related literature

For a related structure, see: Ergenç *et al.* (1992). For general background, see: Jalilian *et al.* (2000); John (1998); Kucukguzel *et al.* (2006); Shen *et al.* (1998); Singh *et al.* (2005). For ring motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

 $\text{C}_{21}\text{H}_{18}\text{ClN}_3\text{O}_2\text{S}$
 $M_r = 411.89$
 Monoclinic, $P2_1/c$
 $a = 14.1039$ (19) Å
 $b = 18.1566$ (19) Å

 $c = 16.9108$ (19) Å
 $\beta = 114.509$ (10)°
 $V = 3940.3$ (9) Å³
 $Z = 8$
 Mo $K\alpha$ radiation

 $\mu = 0.32$ mm⁻¹
 $T = 173$ (2) K

 $0.9 \times 0.4 \times 0.4$ mm

Data collection

 Bruker P4 diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
 $T_{\text{min}} = 0.837$, $T_{\text{max}} = 0.879$
 20863 measured reflections
 9027 independent reflections

 6867 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$
 2 standard reflections every 98 reflections
 intensity decay: none

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.102$
 $S = 1.02$
 9027 reflections
 532 parameters

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.91$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.82$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|----------|-------------|-------------|---------------|
| $\text{O1}-\text{H1O}\cdots\text{S2}^i$ | 0.84 | 2.44 | 3.2242 (13) | 156 |
| $\text{N1}-\text{H1N}\cdots\text{S1}$ | 0.89 (2) | 2.44 (2) | 2.9075 (16) | 113.5 (17) |
| $\text{N2}-\text{H2N}\cdots\text{O5}^{ii}$ | 0.81 (2) | 2.12 (2) | 2.870 (2) | 155 (2) |
| $\text{N3}-\text{H3N}\cdots\text{O5}^{ii}$ | 0.88 (2) | 2.17 (2) | 3.003 (2) | 156.1 (18) |
| $\text{O4}-\text{H4O}\cdots\text{S1}^{iii}$ | 0.84 | 2.51 | 3.2707 (13) | 151 |
| $\text{N4}-\text{H4N}\cdots\text{S2}$ | 0.88 (2) | 2.50 (2) | 2.9569 (16) | 113.5 (17) |
| $\text{N5}-\text{H5N}\cdots\text{O2}^{iv}$ | 0.90 (2) | 1.96 (2) | 2.776 (2) | 149 (2) |
| $\text{N6}-\text{H6N}\cdots\text{O2}^{iv}$ | 0.89 (2) | 2.02 (2) | 2.842 (2) | 154 (2) |

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

Data collection: *XSCANS* (Bruker, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2559).

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supplementary materials

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4-(4-Chlorophenyl)-1-(2-hydroxy-2,2-diphenylacetyl)thiosemicarbazide

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Comment

Thiosemicarbazides have received special interest for their potential biological activities (Kucukguzel *et al.*, 2006; Singh *et al.*, 2005). They have also received considerable attention because of the possibility of their use as intermediates in the synthesis of many biologically active heterocyclic compounds such as 1,2,4-triazole derivatives (Ergenç *et al.*, 1992), 1,3,4-thiadiazoles (Jalilian *et al.*, 2000) and many others. As ligands, thiosemicarbazides are useful bidentate ligands (*S*- and *N*- donors) for transition metal ions and their complexes possess many biological activities (Shen *et al.*, 1998). The title compound was synthesized as an intermediate for biologically active 1,2,4-triazole derivative (Ergenç *et al.*, 1992). We report herein its crystal structure.

The asymmetric unit of the title compound contains two independent thiosemicarbazide molecules (Fig 1), where the bond lengths and angles are almost identical (Table 1). In both molecules, the linking C-N-N-C-N units are delocalized and flattened. The C-S and C-O bonds both show the double bond character, while the C-N and N-N bonds in the linking units imply significant electron delocalization. As a result of conjugation, O2-C14 [1.241 (2) Å] and O5-C35 [1.242 (2) Å] bonds are longer than the normal value of 1.20 Å (John, 1998), while N1-C14 [1.323 (2) Å] and N4-C35 [1.327 (2) Å] bonds are in accordance with the C-N double bond length (1.32 Å; John, 1998) and shorter than the C-N single bond length (1.475 Å; John, 1998). The sum of the bond angles around N1, N2, N3, C14, C15 and N4, N5, N6, C35, C36 atoms are about 360°, which implies sp^2 hybridization for these atoms. The thiourea group is approximately planar. The intramolecular N-H...S hydrogen bonds (Table 2) result in the formation of two five-membered rings (S1/N1/N2/C15/H1N) and (S2/N4/N5/C36/H4N).

In the crystal structure, intermolecular N-H...O hydrogen bonds (Table 2) link the molecules into centrosymmetric dimers (Fig. 2), in which they are also linked to the other dimers via intermolecular O-H...S hydrogen bonds (Table 2) leading to infinite corrugated layers parallel to the *bc* plane through $R_2^2(16)$ ring motifs (Bernstein *et al.*, 1995).

Experimental

The title compound was synthesized according to the literature method (Ergenç *et al.*, 1992) by the reaction of equimolar amounts of 2-hydroxy-2,2-diphenyl- acetohydrazide, (1), and 1-chloro-4-isothiocyanatobenzene, (2), (Fig. 3). Crystals suitable for X-ray analysis were obtained by recrystallization from a methanol solution at room temperature.

Refinement

H1N, H2N, H3N, H4N, H5N and H6N atoms (for NH) were located in difference syntheses and refined isotropically [N-H = 0.81 (2)-0.90 (2) Å and $U_{iso}(H) = 0.032$ (5)-0.046 (7) Å²]. The remaining H atoms were positioned geometrically, with O-H = 0.84 Å (for OH) and C-H = 0.95 Å for aromatic H, respectively, and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C,O)$.

Figures

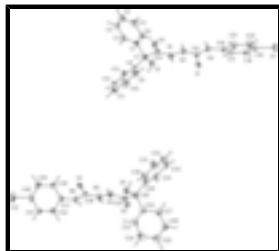


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

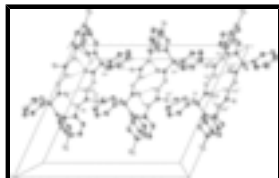


Fig. 2. A partial packing diagram. Hydrogen bonds are shown as dashed lines [symmetry codes: (i) $x, 3/2 - y, z + 1/2$; (ii) $x, y, z + 1$]. H atoms not involved in hydrogen bonding are omitted for clarity.

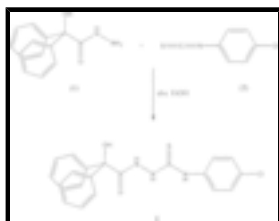


Fig. 3. A schematic representation of the reaction that afforded the title compound.

4-(4-Chlorophenyl)-1-(2-hydroxy-2,2-diphenylacetyl)thiosemicarbazide

Crystal data

$C_{21}H_{18}ClN_3O_2S$

$M_r = 411.89$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 14.1039$ (19) Å

$b = 18.1566$ (19) Å

$c = 16.9108$ (19) Å

$\beta = 114.509$ (10)°

$V = 3940.3$ (9) Å³

$Z = 8$

$F_{000} = 1712$

$D_x = 1.389$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 51 reflections

$\theta = 4.9$ – 12.6 °

$\mu = 0.32$ mm⁻¹

$T = 173$ (2) K

Prism, colorless

$0.9 \times 0.4 \times 0.4$ mm

Data collection

Bruker P4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 173$ (2) K

ω scans

Absorption correction: multi-scan

$R_{int} = 0.029$

$\theta_{max} = 27.5$ °

$\theta_{min} = 2.2$ °

$h = -18 \rightarrow 1$

$k = -23 \rightarrow 23$

$l = -20 \rightarrow 21$

(SADABS; Bruker, 2005)

$T_{\min} = 0.837$, $T_{\max} = 0.879$

20863 measured reflections

9027 independent reflections

6867 reflections with $I > 2\sigma(I)$

2 standard reflections

every 98 reflections

intensity decay: none

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.102$

$S = 1.02$

9027 reflections

532 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2 + 1.5051P]$$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.91 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.82 \text{ e } \text{\AA}^{-3}$

Extinction correction: SHELXL,

$$F_c^* = kFc[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$$

Extinction coefficient: 0.0023 (2)

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|--------------|----------------------------------|
| Cl1 | -0.35372 (4) | 0.47876 (3) | 0.91532 (4) | 0.04719 (14) |
| S1 | 0.10518 (4) | 0.53220 (3) | 0.84004 (3) | 0.03220 (11) |
| O1 | 0.40810 (11) | 0.59866 (7) | 0.86727 (8) | 0.0351 (3) |
| H1O | 0.4136 | 0.5983 | 0.8197 | 0.053* |
| O2 | 0.34635 (10) | 0.74781 (7) | 0.97416 (9) | 0.0372 (3) |
| N1 | 0.26430 (12) | 0.64596 (9) | 0.90301 (10) | 0.0316 (3) |
| H1N | 0.2627 (16) | 0.6047 (12) | 0.8741 (14) | 0.041 (6)* |
| N2 | 0.18196 (12) | 0.65529 (9) | 0.92635 (11) | 0.0327 (4) |
| H2N | 0.1839 (18) | 0.6904 (13) | 0.9562 (15) | 0.046 (7)* |
| N3 | 0.03119 (11) | 0.62184 (8) | 0.93040 (10) | 0.0280 (3) |

supplementary materials

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|-----|---------------|--------------|---------------|--------------|
| H3N | 0.0423 (15) | 0.6629 (11) | 0.9608 (13) | 0.032 (5)* |
| C1 | 0.53250 (13) | 0.66591 (9) | 0.98982 (11) | 0.0265 (4) |
| C2 | 0.59828 (14) | 0.72619 (10) | 1.02190 (12) | 0.0322 (4) |
| H2 | 0.5820 | 0.7714 | 0.9908 | 0.039* |
| C3 | 0.68734 (15) | 0.72099 (12) | 1.09875 (13) | 0.0384 (4) |
| H3 | 0.7314 | 0.7627 | 1.1202 | 0.046* |
| C4 | 0.71225 (16) | 0.65547 (12) | 1.14417 (13) | 0.0428 (5) |
| H4A | 0.7742 | 0.6516 | 1.1961 | 0.051* |
| C5 | 0.64697 (17) | 0.59559 (12) | 1.11394 (14) | 0.0436 (5) |
| H5 | 0.6635 | 0.5507 | 1.1457 | 0.052* |
| C6 | 0.55727 (15) | 0.60058 (10) | 1.03742 (13) | 0.0349 (4) |
| H6 | 0.5124 | 0.5592 | 1.0173 | 0.042* |
| C7 | 0.43300 (14) | 0.67069 (9) | 0.90531 (11) | 0.0281 (4) |
| C8 | 0.44508 (14) | 0.72594 (10) | 0.84178 (11) | 0.0305 (4) |
| C9 | 0.38732 (17) | 0.79023 (13) | 0.81501 (14) | 0.0450 (5) |
| H9 | 0.3358 | 0.8015 | 0.8356 | 0.054* |
| C10 | 0.4044 (2) | 0.83823 (15) | 0.75830 (17) | 0.0616 (7) |
| H10 | 0.3651 | 0.8824 | 0.7409 | 0.074* |
| C11 | 0.47860 (19) | 0.82192 (15) | 0.72685 (15) | 0.0557 (6) |
| H11 | 0.4897 | 0.8546 | 0.6876 | 0.067* |
| C12 | 0.53596 (16) | 0.75824 (13) | 0.75273 (13) | 0.0441 (5) |
| H12 | 0.5866 | 0.7469 | 0.7311 | 0.053* |
| C13 | 0.52029 (15) | 0.71046 (11) | 0.81031 (12) | 0.0355 (4) |
| H13 | 0.5610 | 0.6669 | 0.8285 | 0.043* |
| C14 | 0.34396 (13) | 0.69218 (10) | 0.93070 (11) | 0.0277 (4) |
| C15 | 0.10457 (13) | 0.60501 (9) | 0.90104 (11) | 0.0263 (4) |
| C16 | -0.06058 (13) | 0.58394 (9) | 0.92138 (11) | 0.0263 (4) |
| C17 | -0.08058 (14) | 0.50973 (10) | 0.90017 (11) | 0.0284 (4) |
| H17 | -0.0326 | 0.4809 | 0.8872 | 0.034* |
| C18 | -0.17141 (14) | 0.47832 (10) | 0.89816 (11) | 0.0305 (4) |
| H18 | -0.1858 | 0.4279 | 0.8831 | 0.037* |
| C19 | -0.24083 (13) | 0.51957 (11) | 0.91782 (12) | 0.0317 (4) |
| C20 | -0.22088 (15) | 0.59270 (11) | 0.94035 (14) | 0.0413 (5) |
| H20 | -0.2680 | 0.6209 | 0.9551 | 0.050* |
| C21 | -0.13118 (15) | 0.62458 (11) | 0.94123 (14) | 0.0391 (5) |
| H21 | -0.1178 | 0.6752 | 0.9557 | 0.047* |
| C12 | 0.85383 (5) | 1.00209 (4) | 0.11087 (5) | 0.06182 (19) |
| S2 | 0.40487 (4) | 0.95519 (3) | 0.18449 (3) | 0.03181 (11) |
| O4 | 0.09276 (10) | 0.90499 (7) | 0.15471 (8) | 0.0336 (3) |
| H4O | 0.0816 | 0.9066 | 0.1998 | 0.050* |
| O5 | 0.13097 (10) | 0.75772 (7) | 0.03205 (8) | 0.0342 (3) |
| N4 | 0.22880 (12) | 0.85044 (9) | 0.11506 (11) | 0.0325 (3) |
| H4N | 0.2363 (17) | 0.8894 (12) | 0.1476 (14) | 0.045 (6)* |
| N5 | 0.31033 (12) | 0.83735 (9) | 0.09191 (11) | 0.0362 (4) |
| H5N | 0.2991 (17) | 0.8017 (13) | 0.0518 (14) | 0.046 (6)* |
| N6 | 0.45659 (12) | 0.86864 (9) | 0.07879 (11) | 0.0350 (4) |
| H6N | 0.4369 (17) | 0.8333 (12) | 0.0391 (14) | 0.043 (6)* |
| C22 | -0.04212 (14) | 0.84829 (9) | 0.02956 (11) | 0.0277 (4) |
| C23 | -0.04205 (16) | 0.90432 (10) | -0.02701 (12) | 0.0353 (4) |

| | | | | |
|-----|---------------|--------------|---------------|------------|
| H23 | 0.0192 | 0.9327 | -0.0141 | 0.042* |
| C24 | -0.13058 (17) | 0.91890 (11) | -0.10186 (13) | 0.0418 (5) |
| H24 | -0.1298 | 0.9574 | -0.1396 | 0.050* |
| C25 | -0.22013 (16) | 0.87771 (11) | -0.12200 (13) | 0.0406 (5) |
| H25 | -0.2807 | 0.8877 | -0.1734 | 0.049* |
| C26 | -0.22073 (16) | 0.82195 (11) | -0.06669 (13) | 0.0391 (4) |
| H26 | -0.2819 | 0.7933 | -0.0803 | 0.047* |
| C27 | -0.13221 (14) | 0.80740 (10) | 0.00893 (12) | 0.0319 (4) |
| H27 | -0.1336 | 0.7691 | 0.0467 | 0.038* |
| C28 | 0.05769 (14) | 0.83534 (9) | 0.11184 (11) | 0.0272 (4) |
| C29 | 0.04543 (14) | 0.77964 (10) | 0.17447 (11) | 0.0306 (4) |
| C30 | 0.10095 (18) | 0.71399 (13) | 0.19657 (15) | 0.0487 (5) |
| H30 | 0.1475 | 0.7019 | 0.1709 | 0.058* |
| C31 | 0.0892 (2) | 0.66601 (15) | 0.25556 (18) | 0.0653 (7) |
| H31 | 0.1275 | 0.6213 | 0.2700 | 0.078* |
| C32 | 0.0225 (2) | 0.68284 (15) | 0.29325 (15) | 0.0581 (7) |
| H32 | 0.0146 | 0.6497 | 0.3336 | 0.070* |
| C33 | -0.03331 (17) | 0.74784 (13) | 0.27265 (13) | 0.0466 (5) |
| H33 | -0.0792 | 0.7596 | 0.2990 | 0.056* |
| C34 | -0.02215 (15) | 0.79620 (11) | 0.21312 (12) | 0.0364 (4) |
| H34 | -0.0609 | 0.8408 | 0.1988 | 0.044* |
| C35 | 0.14256 (14) | 0.81022 (10) | 0.08231 (11) | 0.0285 (4) |
| C36 | 0.39154 (14) | 0.88456 (10) | 0.11629 (11) | 0.0293 (4) |
| C37 | 0.55058 (14) | 0.90486 (10) | 0.08987 (11) | 0.0290 (4) |
| C38 | 0.62840 (16) | 0.92071 (13) | 0.17052 (13) | 0.0420 (5) |
| H38 | 0.6183 | 0.9107 | 0.2216 | 0.050* |
| C39 | 0.72165 (16) | 0.95127 (13) | 0.17680 (14) | 0.0473 (5) |
| H39 | 0.7754 | 0.9623 | 0.2322 | 0.057* |
| C40 | 0.73569 (15) | 0.96547 (11) | 0.10254 (13) | 0.0366 (4) |
| C41 | 0.65866 (14) | 0.95032 (10) | 0.02156 (12) | 0.0322 (4) |
| H41 | 0.6691 | 0.9603 | -0.0294 | 0.039* |
| C42 | 0.56583 (14) | 0.92029 (10) | 0.01565 (11) | 0.0298 (4) |
| H42 | 0.5119 | 0.9101 | -0.0399 | 0.036* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|---------------|------------|---------------|
| Cl1 | 0.0334 (3) | 0.0594 (3) | 0.0571 (3) | -0.0161 (2) | 0.0271 (2) | -0.0117 (3) |
| S1 | 0.0354 (2) | 0.0322 (2) | 0.0347 (2) | -0.00651 (19) | 0.0203 (2) | -0.00557 (18) |
| O1 | 0.0414 (8) | 0.0347 (7) | 0.0386 (7) | -0.0121 (6) | 0.0261 (6) | -0.0140 (6) |
| O2 | 0.0322 (7) | 0.0400 (7) | 0.0472 (8) | -0.0083 (6) | 0.0245 (6) | -0.0162 (6) |
| N1 | 0.0269 (8) | 0.0357 (9) | 0.0384 (8) | -0.0060 (7) | 0.0196 (7) | -0.0086 (7) |
| N2 | 0.0273 (8) | 0.0342 (9) | 0.0427 (9) | -0.0073 (7) | 0.0206 (7) | -0.0112 (7) |
| N3 | 0.0254 (8) | 0.0269 (8) | 0.0341 (8) | -0.0040 (6) | 0.0149 (6) | -0.0040 (6) |
| C1 | 0.0246 (8) | 0.0295 (9) | 0.0313 (9) | -0.0011 (7) | 0.0174 (7) | -0.0044 (7) |
| C2 | 0.0311 (10) | 0.0328 (9) | 0.0347 (9) | -0.0040 (8) | 0.0158 (8) | -0.0008 (8) |
| C3 | 0.0307 (10) | 0.0471 (12) | 0.0375 (10) | -0.0097 (9) | 0.0143 (9) | -0.0054 (9) |
| C4 | 0.0317 (10) | 0.0582 (13) | 0.0359 (10) | 0.0033 (10) | 0.0113 (9) | 0.0056 (9) |

supplementary materials

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|-----|-------------|-------------|-------------|--------------|--------------|---------------|
| C5 | 0.0469 (12) | 0.0416 (11) | 0.0457 (12) | 0.0109 (10) | 0.0228 (10) | 0.0114 (9) |
| C6 | 0.0383 (11) | 0.0307 (9) | 0.0421 (10) | -0.0011 (8) | 0.0232 (9) | -0.0011 (8) |
| C7 | 0.0285 (9) | 0.0286 (9) | 0.0319 (9) | -0.0054 (7) | 0.0173 (8) | -0.0073 (7) |
| C8 | 0.0263 (9) | 0.0387 (10) | 0.0259 (8) | -0.0073 (8) | 0.0102 (7) | -0.0034 (7) |
| C9 | 0.0371 (11) | 0.0558 (13) | 0.0453 (12) | 0.0071 (10) | 0.0204 (10) | 0.0132 (10) |
| C10 | 0.0548 (15) | 0.0659 (16) | 0.0648 (16) | 0.0168 (13) | 0.0255 (13) | 0.0318 (13) |
| C11 | 0.0503 (14) | 0.0730 (17) | 0.0456 (13) | -0.0039 (12) | 0.0216 (11) | 0.0225 (12) |
| C12 | 0.0372 (11) | 0.0629 (14) | 0.0365 (10) | -0.0121 (10) | 0.0197 (9) | -0.0011 (10) |
| C13 | 0.0334 (10) | 0.0429 (11) | 0.0352 (10) | -0.0065 (9) | 0.0193 (8) | -0.0045 (8) |
| C14 | 0.0246 (9) | 0.0323 (9) | 0.0285 (9) | -0.0037 (7) | 0.0133 (7) | -0.0026 (7) |
| C15 | 0.0245 (8) | 0.0294 (9) | 0.0245 (8) | -0.0020 (7) | 0.0098 (7) | 0.0023 (7) |
| C16 | 0.0234 (8) | 0.0302 (9) | 0.0257 (8) | -0.0016 (7) | 0.0107 (7) | 0.0027 (7) |
| C17 | 0.0249 (8) | 0.0298 (9) | 0.0310 (9) | -0.0018 (7) | 0.0121 (7) | -0.0002 (7) |
| C18 | 0.0281 (9) | 0.0328 (9) | 0.0297 (9) | -0.0061 (7) | 0.0110 (7) | -0.0015 (7) |
| C19 | 0.0235 (9) | 0.0410 (10) | 0.0316 (9) | -0.0059 (8) | 0.0124 (7) | 0.0008 (8) |
| C20 | 0.0324 (10) | 0.0395 (11) | 0.0607 (13) | 0.0004 (9) | 0.0279 (10) | -0.0055 (10) |
| C21 | 0.0349 (10) | 0.0312 (10) | 0.0577 (13) | -0.0028 (8) | 0.0256 (10) | -0.0059 (9) |
| C12 | 0.0482 (3) | 0.0682 (4) | 0.0875 (4) | -0.0305 (3) | 0.0465 (3) | -0.0349 (3) |
| S2 | 0.0382 (3) | 0.0311 (2) | 0.0292 (2) | -0.0051 (2) | 0.01702 (19) | -0.00241 (18) |
| O4 | 0.0416 (8) | 0.0312 (7) | 0.0379 (7) | -0.0097 (6) | 0.0262 (6) | -0.0099 (5) |
| O5 | 0.0321 (7) | 0.0342 (7) | 0.0421 (7) | -0.0038 (6) | 0.0213 (6) | -0.0107 (6) |
| N4 | 0.0285 (8) | 0.0358 (9) | 0.0408 (9) | -0.0061 (7) | 0.0220 (7) | -0.0108 (7) |
| N5 | 0.0286 (8) | 0.0410 (9) | 0.0469 (10) | -0.0097 (7) | 0.0236 (8) | -0.0147 (8) |
| N6 | 0.0321 (8) | 0.0409 (9) | 0.0383 (9) | -0.0131 (7) | 0.0209 (7) | -0.0153 (7) |
| C22 | 0.0308 (9) | 0.0255 (8) | 0.0333 (9) | 0.0023 (7) | 0.0198 (8) | -0.0028 (7) |
| C23 | 0.0417 (11) | 0.0321 (10) | 0.0403 (10) | -0.0023 (8) | 0.0254 (9) | -0.0006 (8) |
| C24 | 0.0561 (13) | 0.0348 (10) | 0.0388 (11) | 0.0084 (10) | 0.0240 (10) | 0.0056 (8) |
| C25 | 0.0405 (11) | 0.0437 (11) | 0.0367 (10) | 0.0119 (9) | 0.0151 (9) | 0.0004 (9) |
| C26 | 0.0325 (10) | 0.0427 (11) | 0.0410 (11) | 0.0001 (9) | 0.0142 (9) | -0.0042 (9) |
| C27 | 0.0342 (10) | 0.0309 (9) | 0.0346 (9) | -0.0002 (8) | 0.0181 (8) | -0.0008 (8) |
| C28 | 0.0300 (9) | 0.0256 (8) | 0.0320 (9) | -0.0041 (7) | 0.0187 (8) | -0.0048 (7) |
| C29 | 0.0290 (9) | 0.0344 (10) | 0.0292 (9) | -0.0082 (8) | 0.0128 (8) | -0.0026 (7) |
| C30 | 0.0503 (13) | 0.0486 (13) | 0.0564 (13) | 0.0089 (11) | 0.0312 (11) | 0.0164 (10) |
| C31 | 0.0720 (18) | 0.0561 (15) | 0.0774 (18) | 0.0105 (13) | 0.0405 (15) | 0.0305 (13) |
| C32 | 0.0647 (16) | 0.0634 (16) | 0.0495 (13) | -0.0112 (13) | 0.0269 (12) | 0.0180 (12) |
| C33 | 0.0462 (12) | 0.0634 (15) | 0.0388 (11) | -0.0239 (11) | 0.0263 (10) | -0.0105 (10) |
| C34 | 0.0359 (10) | 0.0413 (11) | 0.0362 (10) | -0.0119 (9) | 0.0191 (9) | -0.0072 (8) |
| C35 | 0.0280 (9) | 0.0307 (9) | 0.0305 (9) | -0.0016 (7) | 0.0158 (8) | 0.0004 (7) |
| C36 | 0.0277 (9) | 0.0332 (9) | 0.0281 (9) | -0.0036 (7) | 0.0127 (7) | 0.0005 (7) |
| C37 | 0.0275 (9) | 0.0297 (9) | 0.0328 (9) | -0.0052 (7) | 0.0156 (7) | -0.0037 (7) |
| C38 | 0.0361 (11) | 0.0634 (14) | 0.0296 (9) | -0.0134 (10) | 0.0168 (8) | -0.0048 (9) |
| C39 | 0.0356 (11) | 0.0711 (15) | 0.0358 (10) | -0.0169 (11) | 0.0155 (9) | -0.0188 (10) |
| C40 | 0.0312 (10) | 0.0364 (10) | 0.0499 (11) | -0.0102 (8) | 0.0246 (9) | -0.0121 (9) |
| C41 | 0.0377 (10) | 0.0293 (9) | 0.0376 (10) | 0.0009 (8) | 0.0234 (8) | 0.0019 (7) |
| C42 | 0.0298 (9) | 0.0333 (9) | 0.0261 (8) | 0.0015 (8) | 0.0114 (7) | -0.0005 (7) |

Geometric parameters (Å, °)

C11—C19

1.7407 (18)

C12—C40

1.7445 (19)

| | | | |
|-----------|-------------|------------|-------------|
| S1—C15 | 1.6791 (18) | S2—C36 | 1.6824 (19) |
| O1—C7 | 1.435 (2) | O4—C28 | 1.439 (2) |
| O1—H10 | 0.8400 | O4—H40 | 0.8400 |
| O2—C14 | 1.241 (2) | O5—C35 | 1.242 (2) |
| N1—C14 | 1.323 (2) | N4—C35 | 1.327 (2) |
| N1—N2 | 1.383 (2) | N4—N5 | 1.380 (2) |
| N1—H1N | 0.89 (2) | N4—H4N | 0.88 (2) |
| N2—C15 | 1.349 (2) | N5—C36 | 1.351 (2) |
| N2—H2N | 0.81 (2) | N5—H5N | 0.90 (2) |
| N3—C15 | 1.355 (2) | N6—C36 | 1.345 (2) |
| N3—C16 | 1.417 (2) | N6—C37 | 1.421 (2) |
| N3—H3N | 0.88 (2) | N6—H6N | 0.89 (2) |
| C1—C2 | 1.391 (2) | C22—C27 | 1.385 (2) |
| C1—C6 | 1.394 (3) | C22—C23 | 1.397 (3) |
| C1—C7 | 1.535 (2) | C22—C28 | 1.532 (3) |
| C2—C3 | 1.386 (3) | C23—C24 | 1.386 (3) |
| C2—H2 | 0.9500 | C23—H23 | 0.9500 |
| C3—C4 | 1.380 (3) | C24—C25 | 1.383 (3) |
| C3—H3 | 0.9500 | C24—H24 | 0.9500 |
| C4—C5 | 1.379 (3) | C25—C26 | 1.381 (3) |
| C4—H4A | 0.9500 | C25—H25 | 0.9500 |
| C5—C6 | 1.387 (3) | C26—C27 | 1.392 (3) |
| C5—H5 | 0.9500 | C26—H26 | 0.9500 |
| C6—H6 | 0.9500 | C27—H27 | 0.9500 |
| C7—C8 | 1.530 (2) | C28—C29 | 1.525 (2) |
| C7—C14 | 1.537 (2) | C28—C35 | 1.545 (2) |
| C8—C9 | 1.387 (3) | C29—C30 | 1.390 (3) |
| C8—C13 | 1.398 (3) | C29—C34 | 1.394 (3) |
| C9—C10 | 1.389 (3) | C30—C31 | 1.385 (3) |
| C9—H9 | 0.9500 | C30—H30 | 0.9500 |
| C10—C11 | 1.389 (3) | C31—C32 | 1.372 (4) |
| C10—H10 | 0.9500 | C31—H31 | 0.9500 |
| C11—C12 | 1.374 (3) | C32—C33 | 1.380 (3) |
| C11—H11 | 0.9500 | C32—H32 | 0.9500 |
| C12—C13 | 1.389 (3) | C33—C34 | 1.393 (3) |
| C12—H12 | 0.9500 | C33—H33 | 0.9500 |
| C13—H13 | 0.9500 | C34—H34 | 0.9500 |
| C16—C21 | 1.388 (2) | C37—C38 | 1.380 (3) |
| C16—C17 | 1.393 (2) | C37—C42 | 1.387 (2) |
| C17—C18 | 1.390 (2) | C38—C39 | 1.390 (3) |
| C17—H17 | 0.9500 | C38—H38 | 0.9500 |
| C18—C19 | 1.379 (3) | C39—C40 | 1.375 (3) |
| C18—H18 | 0.9500 | C39—H39 | 0.9500 |
| C19—C20 | 1.378 (3) | C40—C41 | 1.377 (3) |
| C20—C21 | 1.386 (3) | C41—C42 | 1.383 (2) |
| C20—H20 | 0.9500 | C41—H41 | 0.9500 |
| C21—H21 | 0.9500 | C42—H42 | 0.9500 |
| C7—O1—H10 | 109.5 | C28—O4—H40 | 109.5 |
| C14—N1—N2 | 120.78 (16) | C35—N4—N5 | 120.94 (16) |

supplementary materials

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| C14—N1—H1N | 123.6 (14) | C35—N4—H4N | 123.9 (15) |
| N2—N1—H1N | 115.2 (14) | N5—N4—H4N | 114.8 (15) |
| C15—N2—N1 | 119.41 (16) | C36—N5—N4 | 120.32 (16) |
| C15—N2—H2N | 123.1 (17) | C36—N5—H5N | 123.2 (15) |
| N1—N2—H2N | 117.5 (17) | N4—N5—H5N | 115.4 (15) |
| C15—N3—C16 | 130.79 (15) | C36—N6—C37 | 128.29 (16) |
| C15—N3—H3N | 115.2 (13) | C36—N6—H6N | 116.9 (14) |
| C16—N3—H3N | 114.0 (13) | C37—N6—H6N | 114.7 (14) |
| C2—C1—C6 | 118.51 (17) | C27—C22—C23 | 118.62 (17) |
| C2—C1—C7 | 121.52 (16) | C27—C22—C28 | 123.20 (16) |
| C6—C1—C7 | 119.94 (16) | C23—C22—C28 | 118.18 (16) |
| C3—C2—C1 | 120.69 (18) | C24—C23—C22 | 120.55 (18) |
| C3—C2—H2 | 119.7 | C24—C23—H23 | 119.7 |
| C1—C2—H2 | 119.7 | C22—C23—H23 | 119.7 |
| C4—C3—C2 | 120.20 (19) | C25—C24—C23 | 120.42 (19) |
| C4—C3—H3 | 119.9 | C25—C24—H24 | 119.8 |
| C2—C3—H3 | 119.9 | C23—C24—H24 | 119.8 |
| C5—C4—C3 | 119.79 (19) | C26—C25—C24 | 119.42 (19) |
| C5—C4—H4A | 120.1 | C26—C25—H25 | 120.3 |
| C3—C4—H4A | 120.1 | C24—C25—H25 | 120.3 |
| C4—C5—C6 | 120.30 (19) | C25—C26—C27 | 120.40 (19) |
| C4—C5—H5 | 119.9 | C25—C26—H26 | 119.8 |
| C6—C5—H5 | 119.9 | C27—C26—H26 | 119.8 |
| C5—C6—C1 | 120.49 (18) | C22—C27—C26 | 120.58 (18) |
| C5—C6—H6 | 119.8 | C22—C27—H27 | 119.7 |
| C1—C6—H6 | 119.8 | C26—C27—H27 | 119.7 |
| O1—C7—C8 | 111.13 (14) | O4—C28—C29 | 110.30 (14) |
| O1—C7—C1 | 108.97 (14) | O4—C28—C22 | 108.47 (14) |
| C8—C7—C1 | 111.45 (14) | C29—C28—C22 | 113.96 (14) |
| O1—C7—C14 | 106.27 (13) | O4—C28—C35 | 105.77 (13) |
| C8—C7—C14 | 111.82 (15) | C29—C28—C35 | 110.80 (14) |
| C1—C7—C14 | 106.96 (13) | C22—C28—C35 | 107.16 (13) |
| C9—C8—C13 | 118.70 (18) | C30—C29—C34 | 118.53 (18) |
| C9—C8—C7 | 124.01 (17) | C30—C29—C28 | 122.81 (17) |
| C13—C8—C7 | 117.28 (17) | C34—C29—C28 | 118.63 (17) |
| C8—C9—C10 | 120.5 (2) | C31—C30—C29 | 120.7 (2) |
| C8—C9—H9 | 119.8 | C31—C30—H30 | 119.6 |
| C10—C9—H9 | 119.8 | C29—C30—H30 | 119.6 |
| C9—C10—C11 | 120.3 (2) | C32—C31—C30 | 120.3 (2) |
| C9—C10—H10 | 119.8 | C32—C31—H31 | 119.9 |
| C11—C10—H10 | 119.8 | C30—C31—H31 | 119.9 |
| C12—C11—C10 | 119.7 (2) | C31—C32—C33 | 120.1 (2) |
| C12—C11—H11 | 120.2 | C31—C32—H32 | 119.9 |
| C10—C11—H11 | 120.2 | C33—C32—H32 | 119.9 |
| C11—C12—C13 | 120.3 (2) | C32—C33—C34 | 119.9 (2) |
| C11—C12—H12 | 119.8 | C32—C33—H33 | 120.1 |
| C13—C12—H12 | 119.8 | C34—C33—H33 | 120.1 |
| C12—C13—C8 | 120.5 (2) | C33—C34—C29 | 120.5 (2) |
| C12—C13—H13 | 119.7 | C33—C34—H34 | 119.8 |

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| C8—C13—H13 | 119.7 | C29—C34—H34 | 119.8 |
| O2—C14—N1 | 122.43 (16) | O5—C35—N4 | 123.06 (16) |
| O2—C14—C7 | 123.19 (15) | O5—C35—C28 | 123.25 (15) |
| N1—C14—C7 | 114.37 (15) | N4—C35—C28 | 113.68 (15) |
| N2—C15—N3 | 111.80 (15) | N6—C36—N5 | 112.18 (16) |
| N2—C15—S1 | 121.19 (13) | N6—C36—S2 | 125.65 (14) |
| N3—C15—S1 | 127.00 (13) | N5—C36—S2 | 122.17 (14) |
| C21—C16—C17 | 119.19 (16) | C38—C37—C42 | 119.58 (17) |
| C21—C16—N3 | 115.52 (15) | C38—C37—N6 | 122.87 (16) |
| C17—C16—N3 | 125.15 (16) | C42—C37—N6 | 117.42 (16) |
| C18—C17—C16 | 119.30 (17) | C37—C38—C39 | 119.87 (18) |
| C18—C17—H17 | 120.3 | C37—C38—H38 | 120.1 |
| C16—C17—H17 | 120.3 | C39—C38—H38 | 120.1 |
| C19—C18—C17 | 120.72 (17) | C40—C39—C38 | 119.69 (18) |
| C19—C18—H18 | 119.6 | C40—C39—H39 | 120.2 |
| C17—C18—H18 | 119.6 | C38—C39—H39 | 120.2 |
| C20—C19—C18 | 120.42 (17) | C39—C40—C41 | 121.18 (17) |
| C20—C19—C11 | 119.79 (15) | C39—C40—C12 | 119.50 (16) |
| C18—C19—C11 | 119.79 (14) | C41—C40—C12 | 119.31 (15) |
| C19—C20—C21 | 119.10 (18) | C40—C41—C42 | 118.90 (17) |
| C19—C20—H20 | 120.4 | C40—C41—H41 | 120.5 |
| C21—C20—H20 | 120.4 | C42—C41—H41 | 120.5 |
| C20—C21—C16 | 121.25 (18) | C41—C42—C37 | 120.77 (17) |
| C20—C21—H21 | 119.4 | C41—C42—H42 | 119.6 |
| C16—C21—H21 | 119.4 | C37—C42—H42 | 119.6 |
| C14—N1—N2—C15 | -176.39 (17) | C35—N4—N5—C36 | 171.09 (17) |
| C6—C1—C2—C3 | 1.0 (3) | C27—C22—C23—C24 | 0.4 (3) |
| C7—C1—C2—C3 | 179.43 (16) | C28—C22—C23—C24 | -179.29 (16) |
| C1—C2—C3—C4 | 0.5 (3) | C22—C23—C24—C25 | -0.5 (3) |
| C2—C3—C4—C5 | -1.5 (3) | C23—C24—C25—C26 | 0.2 (3) |
| C3—C4—C5—C6 | 1.0 (3) | C24—C25—C26—C27 | 0.3 (3) |
| C4—C5—C6—C1 | 0.5 (3) | C23—C22—C27—C26 | 0.0 (3) |
| C2—C1—C6—C5 | -1.5 (3) | C28—C22—C27—C26 | 179.73 (16) |
| C7—C1—C6—C5 | -179.97 (17) | C25—C26—C27—C22 | -0.4 (3) |
| C2—C1—C7—O1 | 153.48 (15) | C27—C22—C28—O4 | -130.14 (17) |
| C6—C1—C7—O1 | -28.1 (2) | C23—C22—C28—O4 | 49.6 (2) |
| C2—C1—C7—C8 | 30.5 (2) | C27—C22—C28—C29 | -6.9 (2) |
| C6—C1—C7—C8 | -151.15 (16) | C23—C22—C28—C29 | 172.85 (15) |
| C2—C1—C7—C14 | -92.03 (19) | C27—C22—C28—C35 | 116.09 (18) |
| C6—C1—C7—C14 | 86.35 (19) | C23—C22—C28—C35 | -64.21 (19) |
| O1—C7—C8—C9 | 122.7 (2) | O4—C28—C29—C30 | -119.9 (2) |
| C1—C7—C8—C9 | -115.6 (2) | C22—C28—C29—C30 | 117.8 (2) |
| C14—C7—C8—C9 | 4.1 (2) | C35—C28—C29—C30 | -3.1 (2) |
| O1—C7—C8—C13 | -58.3 (2) | O4—C28—C29—C34 | 58.3 (2) |
| C1—C7—C8—C13 | 63.5 (2) | C22—C28—C29—C34 | -64.0 (2) |
| C14—C7—C8—C13 | -176.87 (15) | C35—C28—C29—C34 | 175.04 (15) |
| C13—C8—C9—C10 | -0.2 (3) | C34—C29—C30—C31 | 0.1 (3) |
| C7—C8—C9—C10 | 178.8 (2) | C28—C29—C30—C31 | 178.3 (2) |
| C8—C9—C10—C11 | 0.8 (4) | C29—C30—C31—C32 | -0.1 (4) |

supplementary materials

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|-----------------|--------------|-----------------|--------------|
| C9—C10—C11—C12 | -0.6 (4) | C30—C31—C32—C33 | -0.1 (4) |
| C10—C11—C12—C13 | -0.3 (4) | C31—C32—C33—C34 | 0.3 (4) |
| C11—C12—C13—C8 | 0.9 (3) | C32—C33—C34—C29 | -0.4 (3) |
| C9—C8—C13—C12 | -0.6 (3) | C30—C29—C34—C33 | 0.1 (3) |
| C7—C8—C13—C12 | -179.73 (17) | C28—C29—C34—C33 | -178.10 (17) |
| N2—N1—C14—O2 | -4.2 (3) | N5—N4—C35—O5 | 2.4 (3) |
| N2—N1—C14—C7 | 175.22 (16) | N5—N4—C35—C28 | -176.98 (16) |
| O1—C7—C14—O2 | 171.68 (16) | O4—C28—C35—O5 | -169.03 (16) |
| C8—C7—C14—O2 | -66.9 (2) | C29—C28—C35—O5 | 71.4 (2) |
| C1—C7—C14—O2 | 55.4 (2) | C22—C28—C35—O5 | -53.4 (2) |
| O1—C7—C14—N1 | -7.7 (2) | O4—C28—C35—N4 | 10.4 (2) |
| C8—C7—C14—N1 | 113.68 (17) | C29—C28—C35—N4 | -109.15 (17) |
| C1—C7—C14—N1 | -124.05 (16) | C22—C28—C35—N4 | 125.97 (16) |
| N1—N2—C15—N3 | 179.07 (15) | C37—N6—C36—N5 | -178.74 (18) |
| N1—N2—C15—S1 | -1.2 (2) | C37—N6—C36—S2 | 2.3 (3) |
| C16—N3—C15—N2 | -178.13 (16) | N4—N5—C36—N6 | -171.84 (17) |
| C16—N3—C15—S1 | 2.2 (3) | N4—N5—C36—S2 | 7.1 (3) |
| C15—N3—C16—C21 | -165.17 (18) | C36—N6—C37—C38 | 49.5 (3) |
| C15—N3—C16—C17 | 19.2 (3) | C36—N6—C37—C42 | -134.7 (2) |
| C21—C16—C17—C18 | 0.9 (3) | C42—C37—C38—C39 | -0.6 (3) |
| N3—C16—C17—C18 | 176.41 (16) | N6—C37—C38—C39 | 175.2 (2) |
| C16—C17—C18—C19 | -0.7 (3) | C37—C38—C39—C40 | 0.0 (3) |
| C17—C18—C19—C20 | -0.4 (3) | C38—C39—C40—C41 | 0.3 (3) |
| C17—C18—C19—C11 | -179.70 (14) | C38—C39—C40—C12 | -178.71 (18) |
| C18—C19—C20—C21 | 1.3 (3) | C39—C40—C41—C42 | 0.0 (3) |
| C11—C19—C20—C21 | -179.38 (16) | C12—C40—C41—C42 | 179.06 (14) |
| C19—C20—C21—C16 | -1.1 (3) | C40—C41—C42—C37 | -0.7 (3) |
| C17—C16—C21—C20 | 0.0 (3) | C38—C37—C42—C41 | 1.0 (3) |
| N3—C16—C21—C20 | -175.90 (19) | N6—C37—C42—C41 | -175.05 (17) |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|----------|-------------|-------------|---------------|
| O1—H1O \cdots S2 ⁱ | 0.84 | 2.44 | 3.2242 (13) | 156 |
| N1—H1N \cdots S1 | 0.89 (2) | 2.44 (2) | 2.9075 (16) | 113.5 (17) |
| N2—H2N \cdots O5 ⁱⁱ | 0.81 (2) | 2.12 (2) | 2.870 (2) | 155 (2) |
| N3—H3N \cdots O5 ⁱⁱ | 0.88 (2) | 2.17 (2) | 3.003 (2) | 156.1 (18) |
| O4—H4O \cdots S1 ⁱⁱⁱ | 0.84 | 2.51 | 3.2707 (13) | 151 |
| N4—H4N \cdots S2 | 0.88 (2) | 2.50 (2) | 2.9569 (16) | 113.5 (17) |
| N5—H5N \cdots O2 ^{iv} | 0.90 (2) | 1.96 (2) | 2.776 (2) | 149 (2) |
| N6—H6N \cdots O2 ^{iv} | 0.89 (2) | 2.02 (2) | 2.842 (2) | 154 (2) |

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

Fig. 1

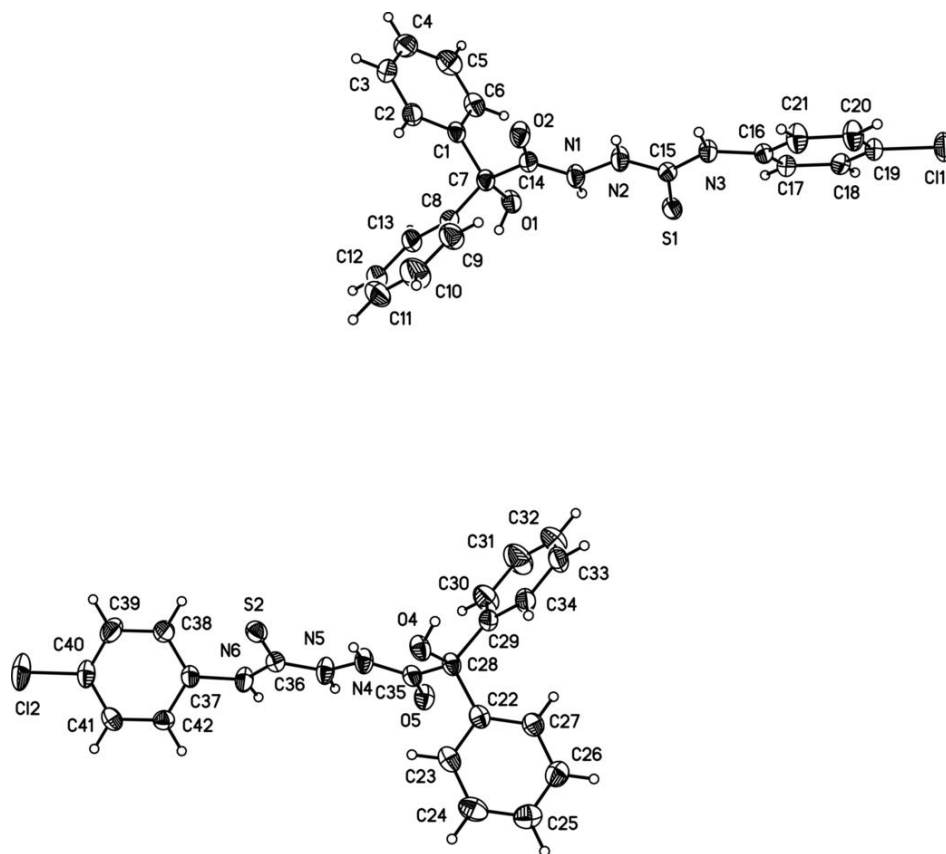


Fig. 2

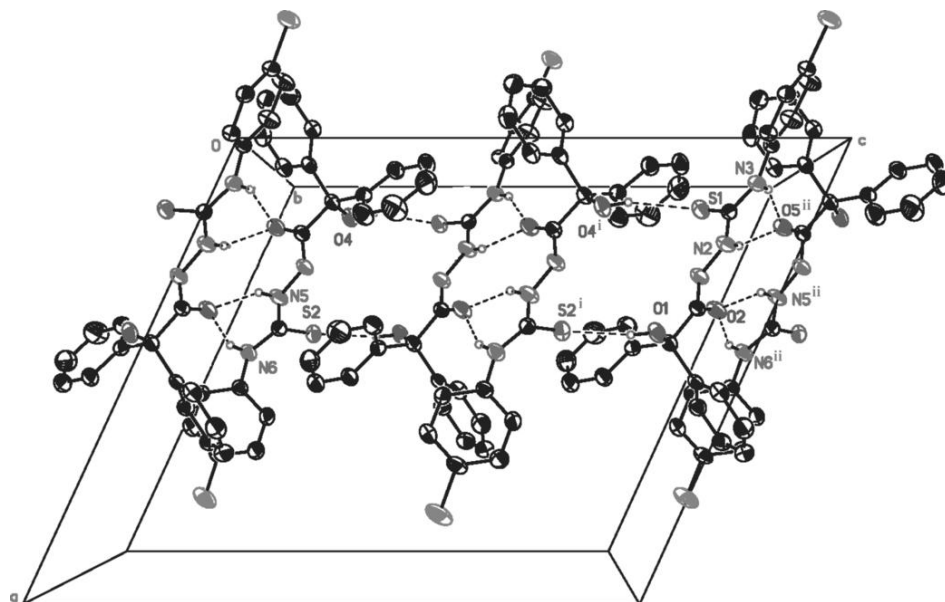


Fig. 3

