

3,3'-Dibutanoyl-1,1'-(*o*-phenylene)dithiourea

Aamer Saeed,^{a*} Naeem Abbas,^a Hummera Rafique^a and Michael Bolte^b

^aDepartment of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan, and ^bInstitut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany

Correspondence e-mail: aamersaeed@yahoo.com

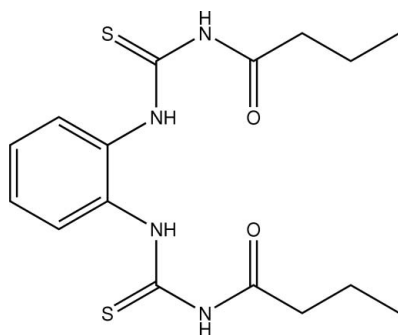
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.036; wR factor = 0.095; data-to-parameter ratio = 14.7.

The molecular conformation of the title compound, $\text{C}_{16}\text{H}_{22}\text{N}_4\text{O}_2\text{S}_2$, is stabilized by two intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. The crystal packing shows $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds.

Related literature

For details of the biological activity of bistioureas, see: Berkessel *et al.* (2006); Moloto *et al.* (2004). For their applications, see: Atia *et al.* (2005); Hu *et al.* (2009); Phetsuksiri *et al.* (2003). For the synthesis of the title compound, see: Succaw *et al.* (2005).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{22}\text{N}_4\text{O}_2\text{S}_2$
 $M_r = 366.50$
 Monoclinic, $P2_1/n$
 $a = 8.8099$ (5) Å

$b = 16.4925$ (7) Å
 $c = 12.3923$ (8) Å
 $\beta = 91.949$ (5)°
 $V = 1799.53$ (17) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.31$ mm⁻¹

$T = 173$ K
 $0.28 \times 0.28 \times 0.23$ mm

Data collection

Stoe IPDS II two-circle diffractometer
 Absorption correction: multi-scan (MULABS; Spek, 2009; Blessing, 1995)
 $T_{\min} = 0.918$, $T_{\max} = 0.932$

22483 measured reflections
 3360 independent reflections
 2890 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.087$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.095$
 $S = 1.04$
 3360 reflections
 229 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.23$ e Å⁻³
 $\Delta\rho_{\min} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|----------|-------------|-------------|---------------|
| $\text{N11}-\text{H11}\cdots\text{O1}$ | 0.86 (2) | 1.90 (2) | 2.6336 (17) | 142.6 (17) |
| $\text{N12}-\text{H12}\cdots\text{O2}^{\dagger}$ | 0.84 (2) | 2.19 (2) | 3.0309 (18) | 175.3 (19) |
| $\text{N21}-\text{H21}\cdots\text{O2}$ | 0.83 (2) | 1.98 (2) | 2.6616 (18) | 139.1 (18) |
| $\text{N22}-\text{H22}\cdots\text{S1}^{\text{ii}}$ | 0.87 (2) | 2.75 (2) | 3.6147 (14) | 172.0 (17) |

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

Data collection: *X-Area* (Stoe & Cie, 2001); cell refinement: *X-Area*; data reduction: *X-Area* (Stoe & Cie, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2318).

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supplementary materials

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3,3'-Dibutanoyl-1,1'-(*o*-phenylene)dithiourea

A. Saeed, N. Abbas, H. Rafique and M. Bolte

Comment

Various bisthiourea derivatives have attracted much attention due to their variety of applications and bioactivities. The presence of multivalent binding sites in bis thioureas provide a multitude of bonding possibilities. Urea and thiourea functionalities, presenting opportunities for the formation of diverse hydrogen bonded networks, represent powerful crystal engineering building blocks (Succaw *et al.*, 2005). The fluorinated bis-thiourea derivative are used as organocatalyst in Morita-Baylis-Hillman reaction (Berkessel *et al.*, 2006). *N*-alkyl thiourea Cadmium(II) complex as precursor for CdS-nanoparticle synthesis (Moloto *et al.*, 2004). BINOL (1,1'-Bi-2-naphthol) bis thiourea derivatives act as chemosensors (Hu *et al.*, 2009). Bis-thiourea resins have been used for adsorption of silver(I) and gold(II) for application to retrieval of silver ions from processed photo films (Atia *et al.*, 2005). Diisoamyloxydiphenylthioureas are effective anti-tuberculosis agents (Phetsuksiri *et al.* (2003).

The molecular conformation of the title compound is stabilized by two N—H···O hydrogen bonds. The crystal packing shows N—H···O and N—H···S hydrogen bonds.

Experimental

The compound was prepared according to literature procedure (Succaw *et al.*, 2005) and Recrystallized from methanol as colourless crystals: Anal. calcd. for C₁₆H₂₂N₄O₂S₂: C, 52.43; H, 6.05; N, 15.29; S, 17.50%; found: C, 52.31; H, 6.19; N, 15.41; S, 17.62.

Refinement

H atoms attached to C were geometrically positioned and refined using a riding model with C—H(aromatic) = 0.95 Å, C—H(methyl) = 0.98 Å, or C—H(methylene) = 0.99 Å, respectively. The position of the amino H atoms were freely refined. In all cases fixed individual displacement parameters

[U(H) = 1.2 U_{eq}(C_{aromatic}), 1.2 U_{eq}(N); 1.5 U_{eq}(C_{methyl})] were used.

Figures

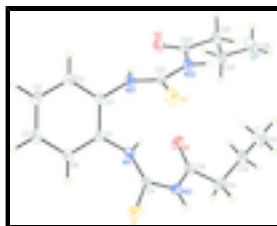


Fig. 1. Molecular structure of title compound. Displacement ellipsoids are drawn at the 50% probability level.

3,3'-Dibutanoyl-1,1'-(*o*-phenylene)dithiourea

Crystal data

| | |
|----------------------------------|---|
| $C_{16}H_{22}N_4O_2S_2$ | $F(000) = 776$ |
| $M_r = 366.50$ | $D_x = 1.353 \text{ Mg m}^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| Hall symbol: -P 2yn | Cell parameters from 20465 reflections |
| $a = 8.8099 (5) \text{ \AA}$ | $\theta = 3.4\text{--}26.0^\circ$ |
| $b = 16.4925 (7) \text{ \AA}$ | $\mu = 0.31 \text{ mm}^{-1}$ |
| $c = 12.3923 (8) \text{ \AA}$ | $T = 173 \text{ K}$ |
| $\beta = 91.949 (5)^\circ$ | Block, colourless |
| $V = 1799.53 (17) \text{ \AA}^3$ | $0.28 \times 0.28 \times 0.23 \text{ mm}$ |
| $Z = 4$ | |

Data collection

| | |
|--|--|
| Stoe IPDS II two-circle diffractometer | 3360 independent reflections |
| Radiation source: fine-focus sealed tube graphite | 2890 reflections with $I > 2\sigma(I)$ |
| ω scans | $R_{\text{int}} = 0.087$ |
| Absorption correction: multi-scan (MULABS; Spek, 2009; Blessing, 1995) | $\theta_{\text{max}} = 25.6^\circ$, $\theta_{\text{min}} = 3.4^\circ$ |
| $T_{\text{min}} = 0.918$, $T_{\text{max}} = 0.932$ | $h = -10 \rightarrow 10$ |
| 22483 measured reflections | $k = -19 \rightarrow 18$ |
| | $l = -15 \rightarrow 14$ |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.036$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.095$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 1.04$ | $w = 1/[\sigma^2(F_o^2) + (0.0595P)^2 + 0.139P]$ |
| 3360 reflections | where $P = (F_o^2 + 2F_c^2)/3$ |
| 229 parameters | $(\Delta/\sigma)_{\text{max}} = 0.001$ |
| 0 restraints | $\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$ |
| | $\Delta\rho_{\text{min}} = -0.33 \text{ e \AA}^{-3}$ |

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> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|--------------|----------------------------------|
| S1 | 0.07444 (5) | 0.68569 (3) | 0.35994 (3) | 0.02446 (14) |
| S2 | 0.47542 (5) | 0.79179 (3) | 0.52094 (3) | 0.02947 (14) |
| O1 | 0.38901 (14) | 0.53284 (9) | 0.57778 (9) | 0.0278 (3) |
| O2 | 0.83408 (13) | 0.59749 (8) | 0.58037 (9) | 0.0224 (3) |
| C1 | 0.42857 (18) | 0.65251 (9) | 0.31239 (12) | 0.0152 (3) |
| C2 | 0.57714 (18) | 0.67920 (9) | 0.33537 (12) | 0.0154 (3) |
| C3 | 0.66576 (19) | 0.70843 (10) | 0.25383 (13) | 0.0206 (3) |
| H3 | 0.7674 | 0.7249 | 0.2695 | 0.025* |
| C4 | 0.6054 (2) | 0.71355 (11) | 0.14871 (13) | 0.0228 (4) |
| H4 | 0.6654 | 0.7342 | 0.0927 | 0.027* |
| C5 | 0.4579 (2) | 0.68847 (10) | 0.12589 (12) | 0.0211 (4) |
| H5 | 0.4170 | 0.6924 | 0.0542 | 0.025* |
| C6 | 0.36860 (19) | 0.65746 (10) | 0.20698 (12) | 0.0180 (3) |
| H6 | 0.2678 | 0.6399 | 0.1906 | 0.022* |
| N11 | 0.34909 (15) | 0.61572 (8) | 0.39740 (10) | 0.0160 (3) |
| H11 | 0.401 (2) | 0.5867 (12) | 0.4430 (16) | 0.019* |
| C11 | 0.20588 (17) | 0.62849 (10) | 0.42462 (12) | 0.0159 (3) |
| N12 | 0.16371 (16) | 0.58921 (8) | 0.51872 (10) | 0.0168 (3) |
| H12 | 0.072 (2) | 0.5945 (12) | 0.5357 (15) | 0.020* |
| C12 | 0.25388 (19) | 0.54519 (10) | 0.59085 (12) | 0.0195 (3) |
| C13 | 0.1723 (2) | 0.51260 (12) | 0.68687 (14) | 0.0272 (4) |
| H13A | 0.1316 | 0.4581 | 0.6691 | 0.033* |
| H13B | 0.0852 | 0.5484 | 0.7017 | 0.033* |
| C14 | 0.2751 (2) | 0.50692 (12) | 0.78795 (14) | 0.0285 (4) |
| H14A | 0.2206 | 0.4776 | 0.8446 | 0.034* |
| H14B | 0.3663 | 0.4748 | 0.7711 | 0.034* |
| C15 | 0.3251 (3) | 0.58888 (14) | 0.83166 (17) | 0.0447 (5) |
| H15A | 0.3912 | 0.5812 | 0.8961 | 0.054* |
| H15B | 0.2357 | 0.6205 | 0.8506 | 0.054* |
| H15C | 0.3809 | 0.6179 | 0.7765 | 0.054* |
| N21 | 0.64067 (16) | 0.67101 (9) | 0.44277 (10) | 0.0170 (3) |
| H21 | 0.708 (2) | 0.6374 (13) | 0.4558 (15) | 0.020* |
| C21 | 0.59630 (17) | 0.71486 (10) | 0.52676 (12) | 0.0171 (3) |
| N22 | 0.65944 (15) | 0.69253 (8) | 0.62711 (11) | 0.0172 (3) |
| H22 | 0.630 (2) | 0.7231 (13) | 0.6794 (16) | 0.021* |
| C22 | 0.77077 (18) | 0.63670 (10) | 0.65070 (12) | 0.0178 (3) |
| C23 | 0.8064 (2) | 0.62499 (11) | 0.76915 (12) | 0.0223 (4) |
| H23A | 0.7462 | 0.6636 | 0.8114 | 0.027* |

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|------|------------|--------------|--------------|------------|
| H23B | 0.9154 | 0.6361 | 0.7847 | 0.027* |
| C24 | 0.7692 (2) | 0.53829 (12) | 0.80244 (14) | 0.0308 (4) |
| H24A | 0.6617 | 0.5264 | 0.7825 | 0.037* |
| H24B | 0.8335 | 0.5000 | 0.7627 | 0.037* |
| C25 | 0.7954 (4) | 0.52563 (15) | 0.92284 (17) | 0.0580 (8) |
| H25A | 0.7694 | 0.4697 | 0.9415 | 0.070* |
| H25B | 0.7314 | 0.5632 | 0.9623 | 0.070* |
| H25C | 0.9024 | 0.5358 | 0.9424 | 0.070* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|---------------|---------------|
| S1 | 0.0188 (2) | 0.0308 (3) | 0.0237 (2) | 0.00497 (16) | 0.00078 (16) | 0.01004 (17) |
| S2 | 0.0334 (3) | 0.0301 (3) | 0.0245 (2) | 0.01694 (19) | -0.00509 (18) | -0.00387 (18) |
| O1 | 0.0216 (6) | 0.0399 (8) | 0.0223 (6) | 0.0105 (5) | 0.0046 (5) | 0.0104 (5) |
| O2 | 0.0225 (6) | 0.0264 (7) | 0.0184 (6) | 0.0085 (5) | -0.0012 (4) | -0.0018 (5) |
| C1 | 0.0168 (7) | 0.0135 (8) | 0.0153 (7) | 0.0021 (6) | 0.0025 (6) | 0.0011 (6) |
| C2 | 0.0181 (8) | 0.0137 (8) | 0.0144 (7) | 0.0028 (6) | -0.0005 (6) | -0.0001 (6) |
| C3 | 0.0182 (8) | 0.0201 (9) | 0.0236 (8) | 0.0003 (6) | 0.0022 (6) | 0.0020 (6) |
| C4 | 0.0268 (9) | 0.0219 (9) | 0.0200 (8) | 0.0017 (7) | 0.0073 (6) | 0.0058 (7) |
| C5 | 0.0289 (9) | 0.0213 (9) | 0.0130 (7) | 0.0052 (7) | 0.0006 (6) | 0.0007 (6) |
| C6 | 0.0191 (8) | 0.0172 (8) | 0.0175 (7) | 0.0012 (6) | -0.0026 (6) | -0.0011 (6) |
| N11 | 0.0152 (6) | 0.0189 (7) | 0.0138 (6) | 0.0005 (5) | -0.0007 (5) | 0.0040 (5) |
| C11 | 0.0175 (8) | 0.0155 (8) | 0.0145 (7) | -0.0034 (6) | -0.0010 (6) | -0.0020 (6) |
| N12 | 0.0137 (7) | 0.0200 (7) | 0.0168 (6) | 0.0006 (5) | 0.0021 (5) | 0.0035 (5) |
| C12 | 0.0216 (8) | 0.0201 (8) | 0.0169 (7) | 0.0026 (6) | 0.0017 (6) | 0.0010 (6) |
| C13 | 0.0256 (9) | 0.0324 (10) | 0.0242 (8) | 0.0047 (7) | 0.0063 (7) | 0.0108 (7) |
| C14 | 0.0391 (10) | 0.0280 (10) | 0.0187 (8) | 0.0083 (8) | 0.0043 (7) | 0.0074 (7) |
| C15 | 0.0663 (16) | 0.0377 (12) | 0.0303 (10) | 0.0012 (11) | 0.0033 (10) | -0.0059 (9) |
| N21 | 0.0153 (7) | 0.0195 (7) | 0.0159 (6) | 0.0045 (5) | -0.0027 (5) | -0.0006 (5) |
| C21 | 0.0147 (7) | 0.0185 (8) | 0.0180 (7) | -0.0001 (6) | 0.0001 (6) | 0.0006 (6) |
| N22 | 0.0190 (7) | 0.0177 (7) | 0.0148 (6) | 0.0015 (5) | -0.0010 (5) | -0.0029 (5) |
| C22 | 0.0186 (8) | 0.0168 (8) | 0.0178 (7) | -0.0021 (6) | -0.0028 (6) | 0.0003 (6) |
| C23 | 0.0296 (9) | 0.0203 (9) | 0.0167 (8) | 0.0036 (7) | -0.0047 (6) | -0.0010 (6) |
| C24 | 0.0442 (11) | 0.0265 (10) | 0.0215 (8) | -0.0072 (8) | 0.0001 (8) | 0.0024 (7) |
| C25 | 0.115 (2) | 0.0344 (13) | 0.0240 (10) | -0.0185 (14) | -0.0110 (12) | 0.0085 (9) |

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

| | | | |
|--------|-------------|----------|-----------|
| S1—C11 | 1.6763 (16) | C13—H13A | 0.9900 |
| S2—C21 | 1.6566 (16) | C13—H13B | 0.9900 |
| O1—C12 | 1.224 (2) | C14—C15 | 1.516 (3) |
| O2—C22 | 1.234 (2) | C14—H14A | 0.9900 |
| C1—C6 | 1.395 (2) | C14—H14B | 0.9900 |
| C1—C2 | 1.401 (2) | C15—H15A | 0.9800 |
| C1—N11 | 1.4207 (19) | C15—H15B | 0.9800 |
| C2—C3 | 1.385 (2) | C15—H15C | 0.9800 |
| C2—N21 | 1.4325 (19) | N21—C21 | 1.337 (2) |
| C3—C4 | 1.393 (2) | N21—H21 | 0.83 (2) |

| | | | |
|---------------|-------------|---------------|-------------|
| C3—H3 | 0.9500 | C21—N22 | 1.395 (2) |
| C4—C5 | 1.384 (3) | N22—C22 | 1.370 (2) |
| C4—H4 | 0.9500 | N22—H22 | 0.87 (2) |
| C5—C6 | 1.394 (2) | C22—C23 | 1.503 (2) |
| C5—H5 | 0.9500 | C23—C24 | 1.527 (3) |
| C6—H6 | 0.9500 | C23—H23A | 0.9900 |
| N11—C11 | 1.334 (2) | C23—H23B | 0.9900 |
| N11—H11 | 0.86 (2) | C24—C25 | 1.516 (3) |
| C11—N12 | 1.395 (2) | C24—H24A | 0.9900 |
| N12—C12 | 1.382 (2) | C24—H24B | 0.9900 |
| N12—H12 | 0.84 (2) | C25—H25A | 0.9800 |
| C12—C13 | 1.510 (2) | C25—H25B | 0.9800 |
| C13—C14 | 1.524 (3) | C25—H25C | 0.9800 |
| C6—C1—C2 | 119.57 (14) | C15—C14—H14B | 108.9 |
| C6—C1—N11 | 122.61 (14) | C13—C14—H14B | 108.9 |
| C2—C1—N11 | 117.65 (13) | H14A—C14—H14B | 107.7 |
| C3—C2—C1 | 120.47 (14) | C14—C15—H15A | 109.5 |
| C3—C2—N21 | 119.91 (14) | C14—C15—H15B | 109.5 |
| C1—C2—N21 | 119.48 (14) | H15A—C15—H15B | 109.5 |
| C2—C3—C4 | 119.80 (15) | C14—C15—H15C | 109.5 |
| C2—C3—H3 | 120.1 | H15A—C15—H15C | 109.5 |
| C4—C3—H3 | 120.1 | H15B—C15—H15C | 109.5 |
| C5—C4—C3 | 119.94 (15) | C21—N21—C2 | 123.85 (14) |
| C5—C4—H4 | 120.0 | C21—N21—H21 | 116.0 (13) |
| C3—C4—H4 | 120.0 | C2—N21—H21 | 120.1 (13) |
| C4—C5—C6 | 120.74 (15) | N21—C21—N22 | 115.68 (14) |
| C4—C5—H5 | 119.6 | N21—C21—S2 | 125.81 (12) |
| C6—C5—H5 | 119.6 | N22—C21—S2 | 118.52 (12) |
| C5—C6—C1 | 119.45 (15) | C22—N22—C21 | 128.97 (14) |
| C5—C6—H6 | 120.3 | C22—N22—H22 | 117.5 (13) |
| C1—C6—H6 | 120.3 | C21—N22—H22 | 113.1 (13) |
| C11—N11—C1 | 127.91 (14) | O2—C22—N22 | 122.64 (14) |
| C11—N11—H11 | 114.1 (12) | O2—C22—C23 | 122.56 (15) |
| C1—N11—H11 | 117.4 (13) | N22—C22—C23 | 114.77 (14) |
| N11—C11—N12 | 114.78 (14) | C22—C23—C24 | 110.15 (14) |
| N11—C11—S1 | 127.73 (12) | C22—C23—H23A | 109.6 |
| N12—C11—S1 | 117.49 (11) | C24—C23—H23A | 109.6 |
| C12—N12—C11 | 128.39 (14) | C22—C23—H23B | 109.6 |
| C12—N12—H12 | 115.2 (13) | C24—C23—H23B | 109.6 |
| C11—N12—H12 | 116.3 (13) | H23A—C23—H23B | 108.1 |
| O1—C12—N12 | 122.84 (14) | C25—C24—C23 | 111.58 (16) |
| O1—C12—C13 | 122.44 (15) | C25—C24—H24A | 109.3 |
| N12—C12—C13 | 114.71 (14) | C23—C24—H24A | 109.3 |
| C12—C13—C14 | 112.57 (15) | C25—C24—H24B | 109.3 |
| C12—C13—H13A | 109.1 | C23—C24—H24B | 109.3 |
| C14—C13—H13A | 109.1 | H24A—C24—H24B | 108.0 |
| C12—C13—H13B | 109.1 | C24—C25—H25A | 109.5 |
| C14—C13—H13B | 109.1 | C24—C25—H25B | 109.5 |
| H13A—C13—H13B | 107.8 | H25A—C25—H25B | 109.5 |

supplementary materials

| | | | |
|-----------------|--------------|-----------------|--------------|
| C15—C14—C13 | 113.35 (16) | C24—C25—H25C | 109.5 |
| C15—C14—H14A | 108.9 | H25A—C25—H25C | 109.5 |
| C13—C14—H14A | 108.9 | H25B—C25—H25C | 109.5 |
| C6—C1—C2—C3 | -1.7 (2) | C11—N12—C12—O1 | -2.4 (3) |
| N11—C1—C2—C3 | 173.81 (15) | C11—N12—C12—C13 | 178.22 (16) |
| C6—C1—C2—N21 | -177.47 (14) | O1—C12—C13—C14 | 31.9 (2) |
| N11—C1—C2—N21 | -2.0 (2) | N12—C12—C13—C14 | -148.67 (15) |
| C1—C2—C3—C4 | 1.9 (2) | C12—C13—C14—C15 | 67.2 (2) |
| N21—C2—C3—C4 | 177.67 (15) | C3—C2—N21—C21 | 113.70 (18) |
| C2—C3—C4—C5 | -0.8 (3) | C1—C2—N21—C21 | -70.5 (2) |
| C3—C4—C5—C6 | -0.4 (3) | C2—N21—C21—N22 | 173.38 (14) |
| C4—C5—C6—C1 | 0.6 (2) | C2—N21—C21—S2 | -6.7 (2) |
| C2—C1—C6—C5 | 0.4 (2) | N21—C21—N22—C22 | 6.5 (2) |
| N11—C1—C6—C5 | -174.83 (15) | S2—C21—N22—C22 | -173.42 (13) |
| C6—C1—N11—C11 | -49.1 (2) | C21—N22—C22—O2 | 1.0 (3) |
| C2—C1—N11—C11 | 135.57 (16) | C21—N22—C22—C23 | -177.18 (15) |
| C1—N11—C11—N12 | -174.00 (14) | O2—C22—C23—C24 | -61.4 (2) |
| C1—N11—C11—S1 | 5.6 (2) | N22—C22—C23—C24 | 116.77 (17) |
| N11—C11—N12—C12 | 6.0 (2) | C22—C23—C24—C25 | -176.8 (2) |
| S1—C11—N12—C12 | -173.65 (13) | | |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|----------|-------------|-------------|---------------|
| N11—H11 \cdots O1 | 0.86 (2) | 1.90 (2) | 2.6336 (17) | 142.6 (17) |
| N12—H12 \cdots O2 ⁱ | 0.84 (2) | 2.19 (2) | 3.0309 (18) | 175.3 (19) |
| N21—H21 \cdots O2 | 0.83 (2) | 1.98 (2) | 2.6616 (18) | 139.1 (18) |
| N22—H22 \cdots S1 ⁱⁱ | 0.87 (2) | 2.75 (2) | 3.6147 (14) | 172.0 (17) |

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

Fig. 1

