

(6-Fluoro-2-oxo-2H-chromen-4-yl)methyl diethyl-carbamodithioate

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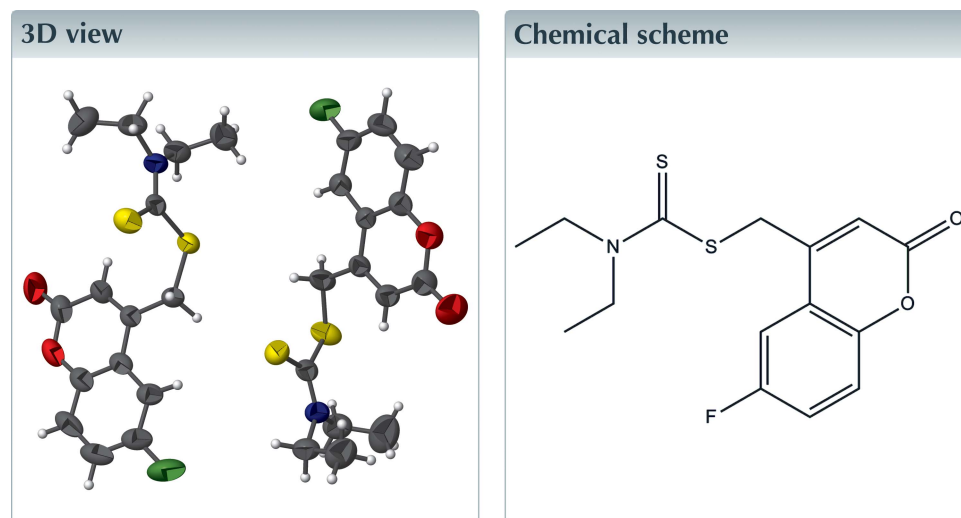
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Keywords: crystal structure; chromene derivatives; carbamodithioate; hydrogen bonding; offset π - π interactions.

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Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{15}H_{16}FNO_2S_2$, crystallizes with two independent molecules (*A* and *B*) in the asymmetric unit. They differ essentially in the orientation of the ethyl groups. The chromene rings are planar (r.m.s. deviations = 0.013 Å for both molecules), with the maximum deviation from the ring planes being 0.014 (2) and 0.018 (2) Å for atoms C9*A* and C9*B*, respectively. The mean plane of the chromene ring makes dihedral angles of 80.01 (7) and 76.97 (8)° with the carbamodithioate moiety [(N-C(=S)-S)] of molecules *A* and *B*, respectively. In the crystal, the two molecules are linked by C-H...S hydrogen bonds, forming a ladder-like arrangement propagating along the *a*-axis direction. Within the ladders there are offset π - π interactions involving the coumarins rings of the *B* molecules [intercentroid distances vary from 3.705 (2) to 3.860 (1) Å]. Neighbouring ladders are linked *via* offset π - π interactions involving the coumarins rings of the *A* molecules [intercentroid distances vary from 3.539 (1) to 3.601 (1) Å]. These latter interactions lead to the formation of layers parallel to the *ac* plane.



Structure description

Coumarins and their derivatives represent an interesting class of heterocyclic compounds, which have attracted attention because of their biological and medicinal properties, such as anti-bacterial (Basanagouda *et al.*, 2009), anti-oxidant (Vukovic *et al.*, 2010) and anti-inflammatory (Emmanuel-Giota *et al.*, 2001). As part of our ongoing studies of coumarin derivatives (El-Khatatneh *et al.*, 2016), the title compound was synthesized and we report herein on its crystal structure.

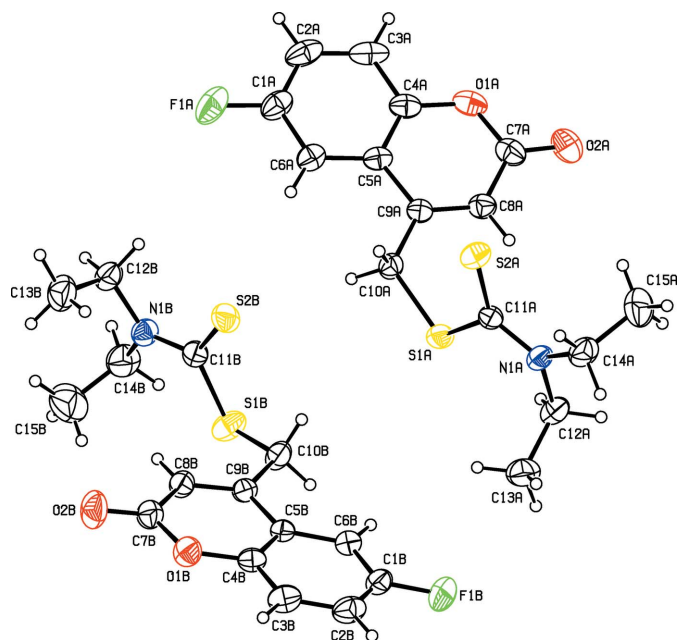


Figure 1
The molecular structure of the two independent molecules of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

The title compound, Fig. 1, crystallized with two independent molecules (*A* and *B*) in the asymmetric unit. The coumarin units are planar with their maximum deviations being 0.014 (2) and 0.018 (2) Å, for atoms C9*A* and C9*B*, respectively. The mean plane of the chromene rings make dihedral angles of 80.01 (7) and 76.97 (8)° with the carbamodithioate moiety [(N–C(=S)–S)] of molecules *A* and *B*, respectively. The AutoMolFit drawing, Fig. 2 (PLATON; Spek, 2009), illustrates that the main difference in the conformation of the two molecules concerns the orientation of the ethyl groups. They are present in *-anti-periplanar* (C14*A*–N1*A*–C11*A*–S1*A*) and *-syn-periplanar* (C14*B*–N1*B*–C11*B*–S1*B*) conformations with respect to the carbamodithioate moiety [(N–C(=S)–S)].

In the crystal, the two molecules are linked by a C–H···S hydrogen bond, and these units are linked by further C–H···S hydrogen bonds, forming a ladder-like arrangement propagating along the *a*-axis direction (Fig. 3 and Table 1).

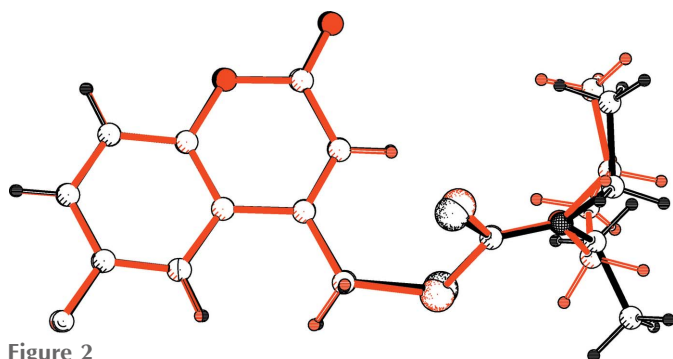


Figure 2
AutoMolFit drawing of molecule *B* (red) on molecule *A* (black) [PLATON; Spek, 2009].

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

| <i>D</i> –H··· <i>A</i> | <i>D</i> –H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> –H··· <i>A</i> |
|--|-------------|---------------|-----------------------|-------------------------|
| C10 <i>A</i> –H10 <i>A</i> ···S2 <i>B</i> | 0.97 | 2.87 | 3.621 (2) | 135 |
| C12 <i>A</i> –H13 <i>C</i> ···O2 <i>B</i> ⁱ | 0.97 | 2.60 | 3.368 (3) | 136 |
| C2 <i>B</i> –H2 <i>B</i> ···S2 <i>B</i> ⁱⁱ | 0.93 | 2.87 | 3.693 (3) | 148 |
| C10 <i>B</i> –H10 <i>C</i> ···S2 <i>A</i> ⁱⁱⁱ | 0.97 | 2.82 | 3.660 (2) | 145 |

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

Table 2
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | C ₁₅ H ₁₆ FNO ₂ S ₂ |
| <i>M</i> _r | 325.41 |
| Crystal system, space group | Triclinic, <i>P</i> $\bar{1}$ |
| Temperature (K) | 293 |
| <i>a</i> , <i>b</i> , <i>c</i> (Å) | 7.1284 (2), 12.2818 (3), 18.3709 (5) |
| α , β , γ (°) | 75.625 (2), 88.538 (2), 86.553 (2) |
| <i>V</i> (Å ³) | 1555.10 (7) |
| <i>Z</i> | 4 |
| Radiation type | Mo <i>K</i> α |
| μ (mm ⁻¹) | 0.36 |
| Crystal size (mm) | 0.30 × 0.25 × 0.20 |
| Data collection | |
| Diffractometer | Bruker APEXII CCD diffractometer |
| No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections | 18765, 4555, 3711 |
| <i>R</i> _{int} | 0.029 |
| θ _{max} (°) | 23.5 |
| (sin θ/λ) _{max} (Å ⁻¹) | 0.560 |
| Refinement | |
| <i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i> | 0.036, 0.094, 1.03 |
| No. of reflections | 4555 |
| No. of parameters | 383 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho$ _{max} , $\Delta\rho$ _{min} (e Å ⁻³) | 0.30, –0.16 |

Computer programs: APEX2 and SAINT (Bruker, 2009), SHELXS2016/6 (Sheldrick, 2008), SHELXL2016/6 (Sheldrick, 2015), Mercury (Macrae *et al.*, 2008) and PLATON (Spek, 2009).

Within the ladders there are offset π – π interactions involving the coumarin rings of the *B* molecules [intercentroid distances vary from 3.705 (2) to 3.860 (1) Å]. Neighbouring ladders are

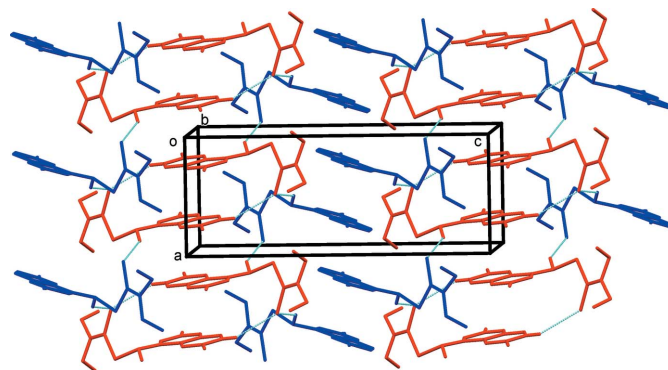


Figure 3
A view along the *b* axis of the crystal packing of the title compound, showing the hydrogen bonds as dashed lines (see Table 1; molecule *A* blue, molecule *B* red). The H atoms not involved in hydrogen bonding have been omitted for clarity.

linked by offset π - π interactions involving the coumarin rings of the *A* molecules [intercentroid distances vary from 3.539 (1) to 3.601 (1) Å], leading to the formation of layers parallel to the *ac* plane (Fig. 3 and Table 1).

Synthesis and crystallization

4-Bromomethyl-6,7-dimethyl-chromen-2-one (3.9 g, 0.015 mol) and the potassium salt of morpholine-4-carboxylate 2.5 g (0.015 mol) were dissolved in 35 ml of absolute ethanol and stirred at room temperature for 14 h. After completion of the reaction (monitored by TLC), the ethanol was removed under reduced pressure. The solid obtained was extracted in ethyl acetate, washed with water, and the collected organic extract was dried over anhydrous Na₂SO₄. The solvent was removed under reduced pressure and the obtained solid product was recrystallized from an ethanol:chloroform mixture (7:3) by slow evaporation, giving colourless block-like crystals.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2017). 2, x162069 [https://doi.org/10.1107/S2414314616020691]

(6-Fluoro-2-oxo-2*H*-chromen-4-yl)methyl diethylcarbamdithioate

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(6-Fluoro-2-oxo-2*H*-chromen-4-yl)methyl diethylcarbamdithioate*Crystal data*

$C_{15}H_{16}FNO_2S_2$

$M_r = 325.41$

Triclinic, $P\bar{1}$

$a = 7.1284$ (2) Å

$b = 12.2818$ (3) Å

$c = 18.3709$ (5) Å

$\alpha = 75.625$ (2)°

$\beta = 88.538$ (2)°

$\gamma = 86.553$ (2)°

$V = 1555.10$ (7) Å³

$Z = 4$

$F(000) = 680$

$D_x = 1.390$ Mg m⁻³

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

Cell parameters from 4557 reflections

$\theta = 1.1$ – 23.5 °

$\mu = 0.36$ mm⁻¹

$T = 293$ K

Block, colourless

$0.30 \times 0.25 \times 0.20$ mm

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: Bruker MicroStar microfocus
rotating anode

Detector resolution: 18.4 pixels mm⁻¹

φ and ω scans

18765 measured reflections

4555 independent reflections

3711 reflections with $I > 2\sigma(I)$

$R_{int} = 0.029$

$\theta_{max} = 23.5$ °, $\theta_{min} = 1.7$ °

$h = -7 \rightarrow 7$

$k = -13 \rightarrow 13$

$l = -20 \rightarrow 20$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.094$

$S = 1.03$

4555 reflections

383 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0413P)^2 + 0.5771P]$

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

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

$\Delta\rho_{max} = 0.30$ e Å⁻³

$\Delta\rho_{min} = -0.16$ e Å⁻³

Special details

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

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|---------------|----------------------------------|
| S1A | 0.51933 (9) | 0.66040 (5) | 0.25174 (3) | 0.05364 (19) |
| S2A | 0.93545 (9) | 0.68645 (6) | 0.22242 (4) | 0.0602 (2) |
| F1A | 0.7749 (3) | 0.18127 (14) | 0.52266 (10) | 0.0948 (6) |
| O1A | 0.7712 (2) | 0.62091 (17) | 0.53152 (9) | 0.0640 (5) |
| O2A | 0.7301 (3) | 0.80267 (19) | 0.48010 (12) | 0.0914 (7) |
| N1A | 0.6629 (3) | 0.83708 (15) | 0.16329 (10) | 0.0512 (5) |
| C1A | 0.7756 (4) | 0.2917 (2) | 0.52380 (16) | 0.0655 (7) |
| C2A | 0.8280 (4) | 0.3182 (3) | 0.58777 (16) | 0.0738 (9) |
| H2A | 0.864836 | 0.261871 | 0.629689 | 0.089* |
| C3A | 0.8258 (3) | 0.4291 (3) | 0.58945 (14) | 0.0683 (8) |
| H3A | 0.860317 | 0.449019 | 0.632678 | 0.082* |
| C4A | 0.7714 (3) | 0.5115 (2) | 0.52580 (13) | 0.0529 (6) |
| C5A | 0.7216 (3) | 0.4849 (2) | 0.46005 (12) | 0.0458 (6) |
| C6A | 0.7236 (3) | 0.3709 (2) | 0.45985 (13) | 0.0531 (6) |
| H6A | 0.690296 | 0.349303 | 0.417061 | 0.064* |
| C7A | 0.7241 (4) | 0.7092 (2) | 0.47157 (15) | 0.0609 (7) |
| C8A | 0.6733 (3) | 0.6818 (2) | 0.40312 (13) | 0.0518 (6) |
| H8A | 0.639944 | 0.740508 | 0.362011 | 0.062* |
| C9A | 0.6715 (3) | 0.57683 (19) | 0.39565 (12) | 0.0431 (5) |
| C10A | 0.6263 (3) | 0.54884 (18) | 0.32320 (12) | 0.0502 (6) |
| H10A | 0.543009 | 0.487043 | 0.334541 | 0.060* |
| H10B | 0.741791 | 0.522131 | 0.302691 | 0.060* |
| C11A | 0.7138 (3) | 0.73768 (18) | 0.20824 (12) | 0.0449 (5) |
| C12A | 0.4672 (4) | 0.8847 (2) | 0.15371 (14) | 0.0628 (7) |
| H13C | 0.466889 | 0.965963 | 0.144464 | 0.075* |
| H13D | 0.396879 | 0.856713 | 0.199792 | 0.075* |
| C13A | 0.3726 (4) | 0.8549 (3) | 0.09011 (16) | 0.0780 (8) |
| H14A | 0.443735 | 0.880548 | 0.044619 | 0.117* |
| H14B | 0.248238 | 0.890242 | 0.084249 | 0.117* |
| H14C | 0.365089 | 0.774673 | 0.100622 | 0.117* |
| C14A | 0.8042 (4) | 0.9084 (2) | 0.11895 (15) | 0.0683 (7) |
| H15A | 0.747783 | 0.953265 | 0.072877 | 0.082* |
| H15B | 0.906248 | 0.861024 | 0.105437 | 0.082* |
| C15A | 0.8818 (5) | 0.9854 (3) | 0.1617 (2) | 0.0991 (11) |
| H16A | 0.781020 | 1.032186 | 0.175377 | 0.149* |
| H16B | 0.971273 | 1.031883 | 0.130680 | 0.149* |
| H16C | 0.942355 | 0.941205 | 0.206303 | 0.149* |
| S1B | 0.08922 (10) | 0.33784 (6) | 0.27010 (4) | 0.0689 (2) |
| S2B | 0.50869 (10) | 0.28748 (6) | 0.28251 (4) | 0.0635 (2) |
| F1B | 0.2068 (3) | 0.76498 (13) | -0.04352 (10) | 0.0965 (6) |
| O1B | 0.2887 (2) | 0.31283 (14) | -0.01045 (9) | 0.0630 (5) |
| O2B | 0.2880 (3) | 0.13736 (16) | 0.05644 (11) | 0.0860 (6) |
| N1B | 0.2507 (3) | 0.15756 (16) | 0.35756 (11) | 0.0536 (5) |
| C1B | 0.2303 (4) | 0.6518 (2) | -0.03385 (16) | 0.0628 (7) |
| C2B | 0.2720 (4) | 0.6108 (3) | -0.09550 (15) | 0.0683 (8) |

| | | | | |
|------|-------------|--------------|---------------|-------------|
| H2B | 0.287050 | 0.659402 | -0.142680 | 0.082* |
| C3B | 0.2911 (4) | 0.4965 (2) | -0.08610 (14) | 0.0642 (7) |
| H3B | 0.318233 | 0.466425 | -0.127166 | 0.077* |
| C4B | 0.2698 (3) | 0.4262 (2) | -0.01553 (13) | 0.0498 (6) |
| C5B | 0.2303 (3) | 0.46786 (18) | 0.04738 (12) | 0.0444 (5) |
| C6B | 0.2095 (3) | 0.5847 (2) | 0.03657 (14) | 0.0554 (6) |
| H6B | 0.181708 | 0.616144 | 0.076986 | 0.067* |
| C7B | 0.2686 (4) | 0.2351 (2) | 0.05671 (15) | 0.0592 (7) |
| C8B | 0.2283 (3) | 0.2784 (2) | 0.12185 (13) | 0.0533 (6) |
| H8B | 0.213405 | 0.227154 | 0.168002 | 0.064* |
| C9B | 0.2111 (3) | 0.38798 (19) | 0.11955 (12) | 0.0467 (6) |
| C10B | 0.1792 (4) | 0.4326 (2) | 0.18835 (13) | 0.0625 (7) |
| H10C | 0.092746 | 0.498300 | 0.175431 | 0.075* |
| H10D | 0.297740 | 0.457461 | 0.201271 | 0.075* |
| C11B | 0.2913 (3) | 0.25121 (19) | 0.30801 (12) | 0.0492 (6) |
| C12B | 0.3993 (4) | 0.0731 (2) | 0.39041 (14) | 0.0639 (7) |
| H13A | 0.354027 | 0.026564 | 0.437468 | 0.077* |
| H13B | 0.506735 | 0.111039 | 0.401320 | 0.077* |
| C13B | 0.4604 (4) | -0.0008 (2) | 0.33904 (19) | 0.0854 (9) |
| H14D | 0.355637 | -0.040849 | 0.329730 | 0.128* |
| H14E | 0.559207 | -0.053535 | 0.362242 | 0.128* |
| H14F | 0.505236 | 0.044952 | 0.292356 | 0.128* |
| C14B | 0.0577 (4) | 0.1330 (3) | 0.38627 (17) | 0.0811 (9) |
| H15C | 0.062020 | 0.100211 | 0.440021 | 0.097* |
| H15D | -0.018925 | 0.202902 | 0.377820 | 0.097* |
| C15B | -0.0283 (5) | 0.0566 (3) | 0.3502 (3) | 0.1224 (14) |
| H16D | -0.049498 | 0.093084 | 0.298224 | 0.184* |
| H16E | -0.146040 | 0.035520 | 0.374573 | 0.184* |
| H16F | 0.053583 | -0.009453 | 0.353828 | 0.184* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| S1A | 0.0565 (4) | 0.0556 (4) | 0.0454 (4) | -0.0069 (3) | -0.0087 (3) | -0.0041 (3) |
| S2A | 0.0540 (4) | 0.0632 (4) | 0.0590 (4) | 0.0101 (3) | -0.0035 (3) | -0.0097 (3) |
| F1A | 0.1031 (13) | 0.0625 (11) | 0.0965 (13) | 0.0007 (9) | 0.0064 (10) | 0.0206 (9) |
| O1A | 0.0563 (11) | 0.0928 (15) | 0.0476 (10) | -0.0056 (10) | -0.0059 (8) | -0.0255 (10) |
| O2A | 0.1197 (18) | 0.0826 (15) | 0.0881 (15) | -0.0025 (13) | -0.0183 (13) | -0.0506 (13) |
| N1A | 0.0553 (12) | 0.0455 (12) | 0.0472 (11) | 0.0008 (9) | -0.0059 (9) | -0.0015 (9) |
| C1A | 0.0523 (16) | 0.0660 (19) | 0.0640 (18) | -0.0004 (13) | 0.0059 (13) | 0.0094 (14) |
| C2A | 0.0516 (16) | 0.091 (2) | 0.0579 (18) | 0.0019 (15) | 0.0000 (13) | 0.0195 (16) |
| C3A | 0.0397 (14) | 0.115 (3) | 0.0403 (14) | -0.0060 (15) | -0.0021 (11) | -0.0011 (15) |
| C4A | 0.0336 (12) | 0.0764 (19) | 0.0464 (14) | -0.0044 (12) | 0.0021 (10) | -0.0112 (13) |
| C5A | 0.0317 (12) | 0.0623 (16) | 0.0395 (13) | -0.0027 (10) | 0.0037 (9) | -0.0057 (11) |
| C6A | 0.0464 (14) | 0.0574 (16) | 0.0490 (14) | -0.0022 (11) | 0.0060 (11) | -0.0016 (12) |
| C7A | 0.0554 (16) | 0.074 (2) | 0.0613 (17) | -0.0032 (13) | -0.0038 (13) | -0.0307 (15) |
| C8A | 0.0528 (14) | 0.0580 (16) | 0.0452 (14) | 0.0011 (12) | -0.0044 (11) | -0.0146 (12) |
| C9A | 0.0369 (12) | 0.0501 (14) | 0.0425 (13) | -0.0020 (10) | 0.0010 (9) | -0.0118 (11) |

| | | | | | | |
|------|-------------|-------------|-------------|--------------|--------------|--------------|
| C10A | 0.0648 (15) | 0.0426 (13) | 0.0411 (13) | -0.0041 (11) | -0.0027 (11) | -0.0062 (10) |
| C11A | 0.0560 (14) | 0.0460 (14) | 0.0349 (12) | -0.0011 (11) | -0.0044 (10) | -0.0142 (10) |
| C12A | 0.0674 (17) | 0.0550 (16) | 0.0600 (16) | 0.0104 (13) | -0.0074 (13) | -0.0053 (13) |
| C13A | 0.0691 (18) | 0.084 (2) | 0.079 (2) | 0.0074 (16) | -0.0180 (15) | -0.0169 (16) |
| C14A | 0.0732 (18) | 0.0617 (17) | 0.0591 (16) | -0.0083 (14) | -0.0017 (14) | 0.0069 (13) |
| C15A | 0.113 (3) | 0.070 (2) | 0.113 (3) | -0.0313 (19) | 0.000 (2) | -0.0137 (19) |
| S1B | 0.0736 (5) | 0.0745 (5) | 0.0507 (4) | 0.0198 (4) | 0.0048 (3) | -0.0070 (3) |
| S2B | 0.0661 (4) | 0.0624 (4) | 0.0603 (4) | -0.0193 (3) | -0.0007 (3) | -0.0083 (3) |
| F1B | 0.1369 (16) | 0.0513 (10) | 0.0896 (12) | -0.0101 (10) | -0.0252 (11) | 0.0084 (9) |
| O1B | 0.0764 (12) | 0.0585 (12) | 0.0570 (11) | 0.0061 (9) | 0.0007 (9) | -0.0222 (9) |
| O2B | 0.1241 (18) | 0.0530 (12) | 0.0868 (15) | 0.0044 (11) | 0.0041 (12) | -0.0310 (11) |
| N1B | 0.0550 (12) | 0.0524 (12) | 0.0496 (12) | -0.0057 (10) | 0.0022 (9) | -0.0049 (10) |
| C1B | 0.0637 (17) | 0.0488 (16) | 0.0674 (19) | -0.0073 (12) | -0.0173 (14) | 0.0045 (14) |
| C2B | 0.0614 (17) | 0.080 (2) | 0.0524 (17) | -0.0100 (15) | -0.0029 (13) | 0.0064 (15) |
| C3B | 0.0597 (16) | 0.080 (2) | 0.0492 (15) | 0.0024 (14) | 0.0032 (12) | -0.0117 (14) |
| C4B | 0.0409 (13) | 0.0590 (16) | 0.0486 (14) | 0.0002 (11) | -0.0027 (10) | -0.0119 (12) |
| C5B | 0.0394 (12) | 0.0443 (14) | 0.0486 (14) | -0.0018 (10) | -0.0085 (10) | -0.0093 (11) |
| C6B | 0.0592 (15) | 0.0508 (15) | 0.0548 (15) | -0.0022 (12) | -0.0127 (12) | -0.0093 (12) |
| C7B | 0.0660 (17) | 0.0523 (17) | 0.0610 (17) | 0.0010 (13) | -0.0027 (13) | -0.0179 (14) |
| C8B | 0.0639 (16) | 0.0459 (15) | 0.0489 (14) | -0.0005 (12) | -0.0024 (12) | -0.0102 (11) |
| C9B | 0.0485 (13) | 0.0459 (14) | 0.0454 (13) | 0.0021 (11) | -0.0061 (10) | -0.0111 (11) |
| C10B | 0.093 (2) | 0.0445 (14) | 0.0479 (14) | 0.0116 (13) | -0.0083 (13) | -0.0102 (12) |
| C11B | 0.0638 (15) | 0.0485 (14) | 0.0370 (12) | -0.0019 (11) | 0.0012 (11) | -0.0142 (11) |
| C12B | 0.0710 (17) | 0.0528 (16) | 0.0603 (16) | -0.0026 (13) | -0.0061 (13) | 0.0008 (13) |
| C13B | 0.087 (2) | 0.0594 (18) | 0.111 (3) | 0.0054 (16) | -0.0021 (19) | -0.0241 (18) |
| C14B | 0.077 (2) | 0.085 (2) | 0.075 (2) | -0.0137 (17) | 0.0029 (16) | -0.0034 (17) |
| C15B | 0.083 (2) | 0.120 (3) | 0.170 (4) | -0.020 (2) | -0.015 (3) | -0.042 (3) |

Geometric parameters (Å, °)

| | | | |
|----------|-----------|----------|-----------|
| S1A—C11A | 1.785 (2) | S1B—C11B | 1.787 (2) |
| S1A—C10A | 1.793 (2) | S1B—C10B | 1.787 (3) |
| S2A—C11A | 1.668 (2) | S2B—C11B | 1.662 (3) |
| F1A—C1A | 1.362 (3) | F1B—C1B | 1.357 (3) |
| O1A—C7A | 1.373 (3) | O1B—C7B | 1.370 (3) |
| O1A—C4A | 1.374 (3) | O1B—C4B | 1.371 (3) |
| O2A—C7A | 1.200 (3) | O2B—C7B | 1.202 (3) |
| N1A—C11A | 1.329 (3) | N1B—C11B | 1.320 (3) |
| N1A—C14A | 1.468 (3) | N1B—C12B | 1.467 (3) |
| N1A—C12A | 1.477 (3) | N1B—C14B | 1.481 (3) |
| C1A—C2A | 1.362 (4) | C1B—C6B | 1.360 (3) |
| C1A—C6A | 1.369 (3) | C1B—C2B | 1.369 (4) |
| C2A—C3A | 1.368 (4) | C2B—C3B | 1.371 (4) |
| C2A—H2A | 0.9300 | C2B—H2B | 0.9300 |
| C3A—C4A | 1.389 (3) | C3B—C4B | 1.378 (3) |
| C3A—H3A | 0.9300 | C3B—H3B | 0.9300 |
| C4A—C5A | 1.387 (3) | C4B—C5B | 1.391 (3) |
| C5A—C6A | 1.400 (3) | C5B—C6B | 1.399 (3) |

| | | | |
|---------------|-------------|---------------|-------------|
| C5A—C9A | 1.453 (3) | C5B—C9B | 1.449 (3) |
| C6A—H6A | 0.9300 | C6B—H6B | 0.9300 |
| C7A—C8A | 1.440 (3) | C7B—C8B | 1.441 (3) |
| C8A—C9A | 1.331 (3) | C8B—C9B | 1.334 (3) |
| C8A—H8A | 0.9300 | C8B—H8B | 0.9300 |
| C9A—C10A | 1.502 (3) | C9B—C10B | 1.504 (3) |
| C10A—H10A | 0.9700 | C10B—H10C | 0.9700 |
| C10A—H10B | 0.9700 | C10B—H10D | 0.9700 |
| C12A—C13A | 1.496 (4) | C12B—C13B | 1.503 (4) |
| C12A—H13C | 0.9700 | C12B—H13A | 0.9700 |
| C12A—H13D | 0.9700 | C12B—H13B | 0.9700 |
| C13A—H14A | 0.9600 | C13B—H14D | 0.9600 |
| C13A—H14B | 0.9600 | C13B—H14E | 0.9600 |
| C13A—H14C | 0.9600 | C13B—H14F | 0.9600 |
| C14A—C15A | 1.507 (4) | C14B—C15B | 1.448 (4) |
| C14A—H15A | 0.9700 | C14B—H15C | 0.9700 |
| C14A—H15B | 0.9700 | C14B—H15D | 0.9700 |
| C15A—H16A | 0.9600 | C15B—H16D | 0.9600 |
| C15A—H16B | 0.9600 | C15B—H16E | 0.9600 |
| C15A—H16C | 0.9600 | C15B—H16F | 0.9600 |
| | | | |
| C11A—S1A—C10A | 103.59 (11) | C11B—S1B—C10B | 103.50 (12) |
| C7A—O1A—C4A | 121.12 (19) | C7B—O1B—C4B | 121.61 (19) |
| C11A—N1A—C14A | 120.6 (2) | C11B—N1B—C12B | 120.9 (2) |
| C11A—N1A—C12A | 124.3 (2) | C11B—N1B—C14B | 123.2 (2) |
| C14A—N1A—C12A | 115.09 (19) | C12B—N1B—C14B | 115.9 (2) |
| C2A—C1A—F1A | 118.9 (2) | F1B—C1B—C6B | 118.3 (3) |
| C2A—C1A—C6A | 123.3 (3) | F1B—C1B—C2B | 118.4 (2) |
| F1A—C1A—C6A | 117.8 (3) | C6B—C1B—C2B | 123.2 (3) |
| C1A—C2A—C3A | 119.0 (3) | C1B—C2B—C3B | 118.5 (2) |
| C1A—C2A—H2A | 120.5 | C1B—C2B—H2B | 120.8 |
| C3A—C2A—H2A | 120.5 | C3B—C2B—H2B | 120.8 |
| C2A—C3A—C4A | 119.2 (3) | C2B—C3B—C4B | 119.7 (3) |
| C2A—C3A—H3A | 120.4 | C2B—C3B—H3B | 120.2 |
| C4A—C3A—H3A | 120.4 | C4B—C3B—H3B | 120.2 |
| O1A—C4A—C5A | 121.9 (2) | O1B—C4B—C3B | 116.5 (2) |
| O1A—C4A—C3A | 116.2 (2) | O1B—C4B—C5B | 121.6 (2) |
| C5A—C4A—C3A | 121.9 (3) | C3B—C4B—C5B | 121.9 (2) |
| C4A—C5A—C6A | 117.8 (2) | C4B—C5B—C6B | 117.6 (2) |
| C4A—C5A—C9A | 118.1 (2) | C4B—C5B—C9B | 118.3 (2) |
| C6A—C5A—C9A | 124.1 (2) | C6B—C5B—C9B | 124.1 (2) |
| C1A—C6A—C5A | 118.8 (3) | C1B—C6B—C5B | 119.1 (2) |
| C1A—C6A—H6A | 120.6 | C1B—C6B—H6B | 120.4 |
| C5A—C6A—H6A | 120.6 | C5B—C6B—H6B | 120.4 |
| O2A—C7A—O1A | 117.6 (2) | O2B—C7B—O1B | 117.5 (2) |
| O2A—C7A—C8A | 125.3 (3) | O2B—C7B—C8B | 125.8 (2) |
| O1A—C7A—C8A | 117.1 (2) | O1B—C7B—C8B | 116.7 (2) |
| C9A—C8A—C7A | 123.4 (2) | C9B—C8B—C7B | 123.6 (2) |

| | | | |
|-----------------|--------------|-----------------|-------------|
| C9A—C8A—H8A | 118.3 | C9B—C8B—H8B | 118.2 |
| C7A—C8A—H8A | 118.3 | C7B—C8B—H8B | 118.2 |
| C8A—C9A—C5A | 118.4 (2) | C8B—C9B—C5B | 118.2 (2) |
| C8A—C9A—C10A | 123.2 (2) | C8B—C9B—C10B | 123.3 (2) |
| C5A—C9A—C10A | 118.3 (2) | C5B—C9B—C10B | 118.5 (2) |
| C9A—C10A—S1A | 116.64 (16) | C9B—C10B—S1B | 116.79 (18) |
| C9A—C10A—H10A | 108.1 | C9B—C10B—H10C | 108.1 |
| S1A—C10A—H10A | 108.1 | S1B—C10B—H10C | 108.1 |
| C9A—C10A—H10B | 108.1 | C9B—C10B—H10D | 108.1 |
| S1A—C10A—H10B | 108.1 | S1B—C10B—H10D | 108.1 |
| H10A—C10A—H10B | 107.3 | H10C—C10B—H10D | 107.3 |
| N1A—C11A—S2A | 124.47 (18) | N1B—C11B—S2B | 124.09 (19) |
| N1A—C11A—S1A | 113.32 (17) | N1B—C11B—S1B | 113.70 (18) |
| S2A—C11A—S1A | 122.20 (13) | S2B—C11B—S1B | 122.22 (14) |
| N1A—C12A—C13A | 112.1 (2) | N1B—C12B—C13B | 112.3 (2) |
| N1A—C12A—H13C | 109.2 | N1B—C12B—H13A | 109.1 |
| C13A—C12A—H13C | 109.2 | C13B—C12B—H13A | 109.1 |
| N1A—C12A—H13D | 109.2 | N1B—C12B—H13B | 109.1 |
| C13A—C12A—H13D | 109.2 | C13B—C12B—H13B | 109.1 |
| H13C—C12A—H13D | 107.9 | H13A—C12B—H13B | 107.9 |
| C12A—C13A—H14A | 109.5 | C12B—C13B—H14D | 109.5 |
| C12A—C13A—H14B | 109.5 | C12B—C13B—H14E | 109.5 |
| H14A—C13A—H14B | 109.5 | H14D—C13B—H14E | 109.5 |
| C12A—C13A—H14C | 109.5 | C12B—C13B—H14F | 109.5 |
| H14A—C13A—H14C | 109.5 | H14D—C13B—H14F | 109.5 |
| H14B—C13A—H14C | 109.5 | H14E—C13B—H14F | 109.5 |
| N1A—C14A—C15A | 111.8 (2) | C15B—C14B—N1B | 112.1 (3) |
| N1A—C14A—H15A | 109.3 | C15B—C14B—H15C | 109.2 |
| C15A—C14A—H15A | 109.3 | N1B—C14B—H15C | 109.2 |
| N1A—C14A—H15B | 109.3 | C15B—C14B—H15D | 109.2 |
| C15A—C14A—H15B | 109.3 | N1B—C14B—H15D | 109.2 |
| H15A—C14A—H15B | 107.9 | H15C—C14B—H15D | 107.9 |
| C14A—C15A—H16A | 109.5 | C14B—C15B—H16D | 109.5 |
| C14A—C15A—H16B | 109.5 | C14B—C15B—H16E | 109.5 |
| H16A—C15A—H16B | 109.5 | H16D—C15B—H16E | 109.5 |
| C14A—C15A—H16C | 109.5 | C14B—C15B—H16F | 109.5 |
| H16A—C15A—H16C | 109.5 | H16D—C15B—H16F | 109.5 |
| H16B—C15A—H16C | 109.5 | H16E—C15B—H16F | 109.5 |
| F1A—C1A—C2A—C3A | -178.9 (2) | F1B—C1B—C2B—C3B | -178.3 (2) |
| C6A—C1A—C2A—C3A | 1.3 (4) | C6B—C1B—C2B—C3B | 1.0 (4) |
| C1A—C2A—C3A—C4A | -0.4 (4) | C1B—C2B—C3B—C4B | -0.6 (4) |
| C7A—O1A—C4A—C5A | -1.0 (3) | C7B—O1B—C4B—C3B | -179.3 (2) |
| C7A—O1A—C4A—C3A | 178.5 (2) | C7B—O1B—C4B—C5B | 0.4 (3) |
| C2A—C3A—C4A—O1A | 179.6 (2) | C2B—C3B—C4B—O1B | 179.3 (2) |
| C2A—C3A—C4A—C5A | -0.9 (4) | C2B—C3B—C4B—C5B | -0.4 (4) |
| O1A—C4A—C5A—C6A | -179.19 (19) | O1B—C4B—C5B—C6B | -178.7 (2) |
| C3A—C4A—C5A—C6A | 1.4 (3) | C3B—C4B—C5B—C6B | 1.1 (3) |

| | | | |
|--------------------|--------------|--------------------|--------------|
| O1A—C4A—C5A—C9A | 1.1 (3) | O1B—C4B—C5B—C9B | 0.6 (3) |
| C3A—C4A—C5A—C9A | -178.3 (2) | C3B—C4B—C5B—C9B | -179.7 (2) |
| C2A—C1A—C6A—C5A | -0.9 (4) | F1B—C1B—C6B—C5B | 179.0 (2) |
| F1A—C1A—C6A—C5A | 179.4 (2) | C2B—C1B—C6B—C5B | -0.3 (4) |
| C4A—C5A—C6A—C1A | -0.5 (3) | C4B—C5B—C6B—C1B | -0.7 (3) |
| C9A—C5A—C6A—C1A | 179.2 (2) | C9B—C5B—C6B—C1B | -179.9 (2) |
| C4A—O1A—C7A—O2A | -178.7 (2) | C4B—O1B—C7B—O2B | -179.8 (2) |
| C4A—O1A—C7A—C8A | 0.6 (3) | C4B—O1B—C7B—C8B | -0.5 (3) |
| O2A—C7A—C8A—C9A | 178.8 (3) | O2B—C7B—C8B—C9B | 178.8 (3) |
| O1A—C7A—C8A—C9A | -0.5 (4) | O1B—C7B—C8B—C9B | -0.3 (4) |
| C7A—C8A—C9A—C5A | 0.7 (3) | C7B—C8B—C9B—C5B | 1.3 (4) |
| C7A—C8A—C9A—C10A | -177.4 (2) | C7B—C8B—C9B—C10B | -176.6 (2) |
| C4A—C5A—C9A—C8A | -0.9 (3) | C4B—C5B—C9B—C8B | -1.4 (3) |
| C6A—C5A—C9A—C8A | 179.4 (2) | C6B—C5B—C9B—C8B | 177.8 (2) |
| C4A—C5A—C9A—C10A | 177.2 (2) | C4B—C5B—C9B—C10B | 176.6 (2) |
| C6A—C5A—C9A—C10A | -2.5 (3) | C6B—C5B—C9B—C10B | -4.2 (3) |
| C8A—C9A—C10A—S1A | -14.5 (3) | C8B—C9B—C10B—S1B | -19.8 (3) |
| C5A—C9A—C10A—S1A | 167.51 (16) | C5B—C9B—C10B—S1B | 162.27 (17) |
| C11A—S1A—C10A—C9A | 81.97 (19) | C11B—S1B—C10B—C9B | 79.0 (2) |
| C14A—N1A—C11A—S2A | 2.9 (3) | C12B—N1B—C11B—S2B | -4.5 (3) |
| C12A—N1A—C11A—S2A | -176.84 (18) | C14B—N1B—C11B—S2B | 172.5 (2) |
| C14A—N1A—C11A—S1A | -175.79 (17) | C12B—N1B—C11B—S1B | 175.80 (17) |
| C12A—N1A—C11A—S1A | 4.4 (3) | C14B—N1B—C11B—S1B | -7.3 (3) |
| C10A—S1A—C11A—N1A | -167.43 (16) | C10B—S1B—C11B—N1B | -163.79 (17) |
| C10A—S1A—C11A—S2A | 13.81 (17) | C10B—S1B—C11B—S2B | 16.45 (18) |
| C11A—N1A—C12A—C13A | -91.7 (3) | C11B—N1B—C12B—C13B | -80.5 (3) |
| C14A—N1A—C12A—C13A | 88.5 (3) | C14B—N1B—C12B—C13B | 102.4 (3) |
| C11A—N1A—C14A—C15A | -89.1 (3) | C11B—N1B—C14B—C15B | 101.2 (3) |
| C12A—N1A—C14A—C15A | 90.7 (3) | C12B—N1B—C14B—C15B | -81.8 (3) |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------------|-------|-------------|-------------|---------------|
| C10A—H10A \cdots S2B | 0.97 | 2.87 | 3.621 (2) | 135 |
| C12A—H13C \cdots O2B ⁱ | 0.97 | 2.60 | 3.368 (3) | 136 |
| C2B—H2B \cdots S2B ⁱⁱ | 0.93 | 2.87 | 3.693 (3) | 148 |
| C10B—H10C \cdots S2A ⁱⁱⁱ | 0.97 | 2.82 | 3.660 (2) | 145 |

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