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# Crystal structures of 6a,6b,7,11a-tetrahydro-6*H*,9*H*-spiro[chromeno[3′,4′:3,4]pyrrolo[1,2-c]-thiazole-11,3′-indoline]-2′,6-dione and 5′-methyl-6a,6b,7,11a-tetrahydro-6*H*,9*H*-spiro[chromeno-[3′,4′:3,4]pyrrolo[1,2-c]thiazole-11,3′-indoline]-2′,6-dione

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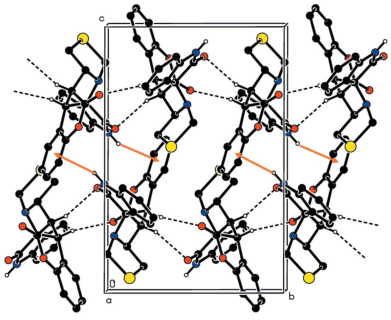
**CCDC references**: 1888373; 1888372

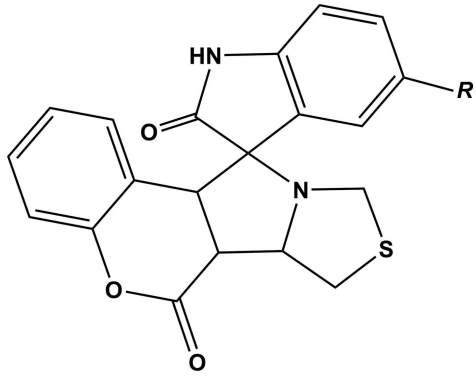
**Supporting information**: this article has supporting information at journals.iucr.org/e

The title compounds, $C_{20}H_{16}N_2O_3S$, (I), and $C_{21}H_{18}N_2O_3S$, (II), differ by the presence of a methyl group in position 5 on the 1*H*-indole-2-one ring of compound (II). The two compounds have a structural overlap r.m.s. deviation of 0.48 Å. There is a significant difference in the conformation of the thiazolidine ring: it has a twisted conformation on the fused N—C bond in (I), but an envelope conformation in compound (II) with the S atom as the flap. The planar pyrrolidine ring of the indole ring system is normal to the mean plane of the five-membered pyrrolidine ring of the pyrrolothiazole unit in both compounds, with dihedral angles of 88.71 (9) and 84.59 (8)°. The pyran rings in both structures have envelope conformations with the methylene C atom adjacent to the C=O group as the flap. In both compounds, there is a short intramolecular C—H···O contact present. In the crystal of (I), molecules are linked by C—H···O hydrogen bonds forming chains propagating along the *b*-axis direction. The chains are linked by N—H···$\pi$ interactions, forming layers parallel to (10$\bar{1}$). In the crystal of (II), molecules are linked by pairs of N—H···O hydrogen bonds, forming inversion dimers which are linked by C—H···O hydrogen bonds to form a three-dimensional structure.

## 1. Chemical context

Indole derivatives have been reported to exhibit a large number of biological activities, such as anti-inflammatory (Chen *et al.*, 2017), anti-fungal (Singh *et al.*, 2000), anti-hepatitis B virus (Chai *et al.*, 2006) and anti-HIV (Sriram *et al.*, 2006; Pandeya *et al.*, 2000). Indole analogues play a significant role in a diverse array of products, such as vitamin supplements, dyes, plastics, flavour enhancers, and in the agricultural and perfumery industries (Barden, 2011). In view of the importance of such compounds, we report herein on the synthesis and molecular and crystal structures of the title compounds, 6a,6b,7,11a-tetrahydro-6*H*,9*H*-spiro[chromeno-[3′,4′:3,4]pyrrolo [1,2-*c*]thiazole-11,3′-indoline]-2′,6-dione (I) and 5′-methyl-6a,6b,7,11a-tetrahydro-6*H*,9*H*-spiro[chromeno-[3′,4′:3,4]pyrrolo [1,2-*c*]thiazole-11,3′-indoline]-2′,6-dione (II).






(I) $R$ = H
(II) $R$ = $CH_3$

## 2. Structural commentary

The molecular structure of compound (I) is illustrated in Fig. 1, and for compound (II) in Fig. 2. The conformations of the two molecules differ by an r.m.s. deviation of 0.48 Å, as shown in the structural overlap figure (Fig. 3). The molecular structures of both compounds are influenced by a short intramolecular C—H···O contact (Tables 1 and 2), which forms an $S$(5) ring motif (Figs. 1 and 2).

There is a significant difference in the conformation of the five-membered thiazolidine ring in the two compounds. In compound (I), the thiazolidine ring (S1/N1/C10–C12) adopts a twist conformation on the N1—C10 bond [$\Delta C_2$(S1) asymmetry parameter is 0.006 (1)]. In (II) this ring adopts an envelope conformation [puckering parameters $q_2$ = 0.529 (2) Å and $\varphi$ = 105.8 (1)°] with atom S1 as the flap, deviating by 0.896 (1) Å from the mean plane through the remaining four atoms.

In compound (I), the pyrrolidine ring (C8–C10/N1/C13) adopts an envelope conformation [puckering parameters $q_2$ = 0.335 (2) Å and $\varphi$ = 39.4 (1)°] with atom C9 as the flap,

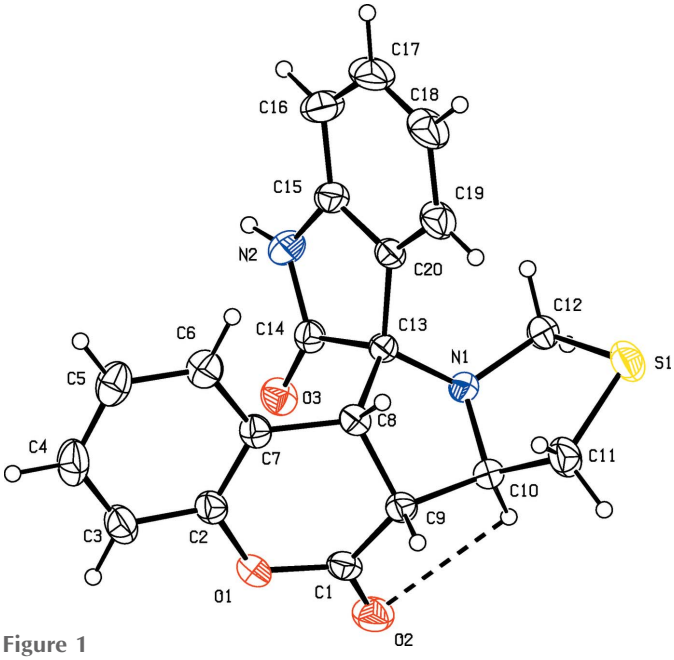



**Figure 1**
A view of the molecular structure of compound (I), showing the atom labelling. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular C—H···O interaction (Table 1) is shown as a dashed line.

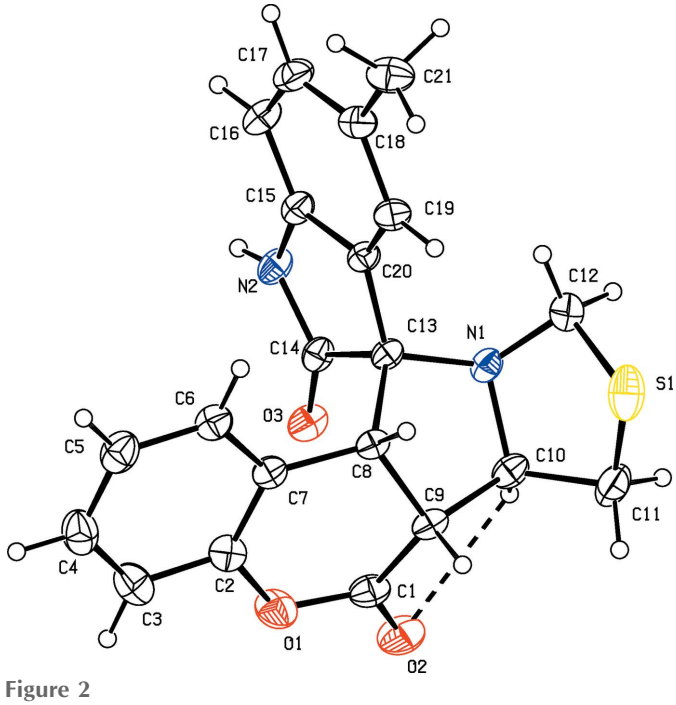



**Figure 2**
A view of the molecular structure of compound (II), showing the atom labelling. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular C—H···O interaction (Table 2) is shown as a dashed line.

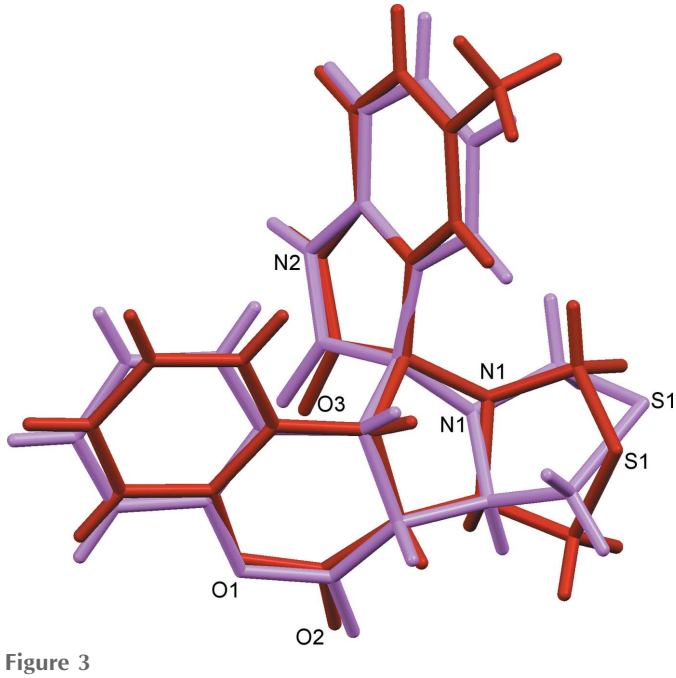



**Figure 3**
Structural overlay of compound (I) (purple) and compound (II) (red). The r.m.s. deviation is 0.48 Å (*Mercury*; Macrae *et al.*, 2008).

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

*Cg* is the centroid of the C2–C7 ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C10—H10···O2 | 0.98 | 2.50 | 2.902 (2) | 104 |
| N2—H2···*Cg*$^{i}$ | 0.86 | 2.57 | 3.799 (18) | 157 |
| C8—H8···O2$^{ii}$ | 0.98 | 2.38 | 3.321 (2) | 160 |
| C9—H9···O3$^{ii}$ | 0.98 | 2.44 | 3.376 (2) | 159 |

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

**Table 2**
Hydrogen-bond geometry (Å, °) for (II).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C10—H10···O2 | 0.98 | 2.44 | 2.882 (2) | 107 |
| N2—H2···O3$^{i}$ | 0.86 | 2.06 | 2.903 (2) | 168 |
| C3—H3···O1$^{ii}$ | 0.93 | 2.55 | 3.302 (2) | 139 |
| C9—H9···O2$^{iii}$ | 0.98 | 2.59 | 3.320 (2) | 131 |
| C21—H21*C*···O2$^{iv}$ | 0.96 | 2.57 | 3.390 (2) | 144 |

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

deviating by 0.518 (2) Å from the mean plane through the remaining four atoms. In (II) this ring adopts a twist conformation on the C8—C13 bond [$\Delta C_2$(C10) asymmetry parameter is 0.005 (1)].

The 2,3-dihydro-1*H*-indol-2-one ring is planar in both compounds, with a maximum deviation of 0.054 (1) and 0.080 (1) Å from the mean plane for atom C14 in (I) and (II), respectively. Oxygen atom O3 of this ring deviates by 0.151 (1) and 0.185 (1) Å, respectively, from the above mean planes. The methyl atom C21 in (II) deviates by 0.056 (2) Å from the plane of the benzene ring to which it is attached.

The pyran rings (C1/O1/C2/C7–C9) in both structures have distorted sofa conformations, with $\Delta C_s$(C2) asymmetry parameters (Nardelli, 1983) of 0.005 (1) and 0.006 (1), respectively. Atom C9 deviates from the mean plane through the remaining five atoms (O1/C1/C2/C7/C8) of the pyran ring by 0.465 (2) Å in (I) and by 0.383 (2) Å in (II).

In both compounds, the planar pyrrolidine ring (N2/C13–C15/C20) of the indole ring system is normal to the mean plane of the pyrrolidine ring (N1/C8–C10/C13) of the pyrrolothiazole unit, with a dihedral angle of 88.71 (9)° for (I) and 84.59 (8)° for (II). The mean plane of the pyrrolidine ring (N1/C8–C10/C13) is inclined to the mean plane of the thiazolidine ring (S1/N1/C10–C12) by 64.39 (2)° in (I) and 79.51 (9)° in (II).

## 3. Supramolecular features

In the crystal of compound (I), molecules associate *via* two C—H···O intermolecular interactions (C8—H8···O2$^{ii}$, C9—H9···O3$^{ii}$, Table 1) forming chains propagating along [001]; see Fig. 4. In addition to this, inversion-related molecules are linked to form dimers by N—H···$\pi$ interactions; N2—H2···*Cg*$^{i}$, where *Cg* is the centroid of the benzene ring (C2–C7); see Fig. 4 and Table 1. The result of these interactions is the formation of layers lying parallel to the (10$\bar{1}$) plane.

In the crystal of compound (II), molecules are linked *via* pairs of N—H···O hydrogen bonds (N2—H2···O3$^{i}$, Table 2), forming inversion dimers with an $R_2^2(8)$ ring motif (Fig. 5). There are two pairs of weak C—H···O intermolecular interactions (C3—H3···O1$^{ii}$, C9—H9···O2$^{iii}$, Table 2) also forming inversion dimers and enclosing $R_2^2(8)$ ring motifs. These dimers are linked to form a helix along the *a*-axis direction. A further C—H···O hydrogen bond (C21—H21*C*···O2$^{iv}$, Table 2) links the molecules to form *C*(10) chains propagating along [010] in

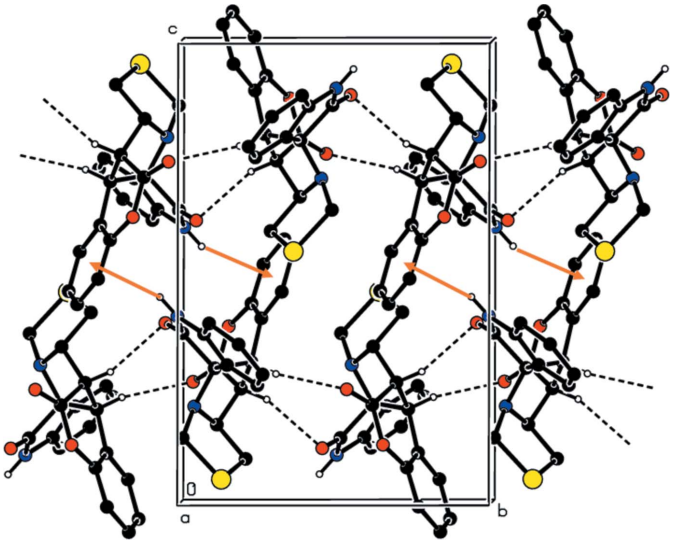



**Figure 4**
The crystal packing of compound (I) viewed along the *a* axis. The C—H···O hydrogen bonds (see Table 1) are shown as dashed lines, while the N—H···$\pi$ interactions are shown as orange arrows. For clarity, H atoms not involved in these interactions have been omitted.

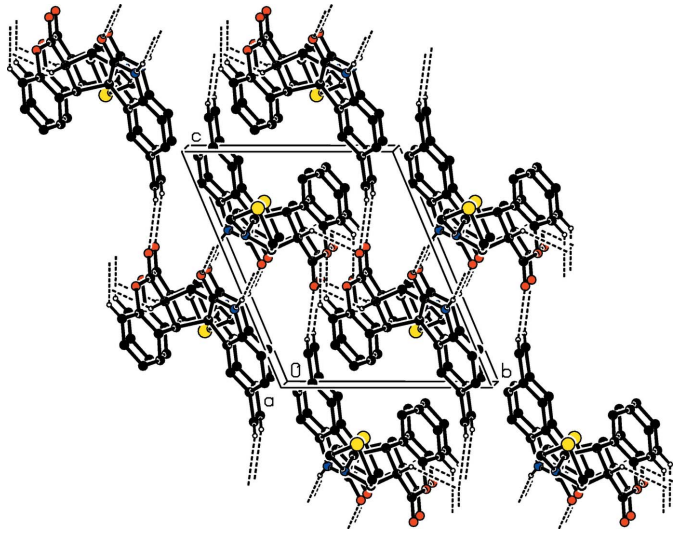



**Figure 5**
The crystal packing of compound (II) viewed along the *a* axis. The N—H···O and C—H···O hydrogen bonds (Table 2) are shown as dashed lines. For clarity, H atoms not involved in the hydrogen bonds have been omitted.

an anti-parallel manner. As a result of the various N—H···O and C—H···O hydrogen bonds, a three-dimensional structure is formed (Table 2 and Fig. 5)

## 4. Database survey

A search of the Cambridge Structural Database (Version 5.39, last update August 2018; Groom *et al.*, 2016) for partial structure *S*1 (Fig. 6) gave three hits. Details are given in the supporting information (CSD search S1). They include: 2,4-dichloro-5′-methyl-6a,6b,7,8,9,11a-hexahydro-6*H*-spiro-[chromeno[3,4-*a*]pyrrolizine-11,3′-indole]-2′,6(1′*H*)-dione monohydrate (GUCGIN; Kanchithalaivan *et al.*, 2014*a*), 3a-acetyl-2-methyl-2,3,3a,9b-tetrahydro-4*H*-spiro[chromeno-[3,4-*c*]pyrrole-1,3′-indole]-2′,4(1′*H*)-dione (SUTLAV; Ghandi *et al.*, 2010), and 8-bromo-2-methyl-2,3,3a,9b-tetrahydro-4*H*-spiro[chromeno[3,4-*c*]pyrrole-1,3′-indole]-2′,4(1′*H*)-dione (SUTLEZ; Ghandi *et al.*, 2010). Here the dihedral angle between the planar pyrrolidine ring of the indole ring system and the mean plane of the pyrrolidine ring of the pyrrolothiazole unit are 82.85, 87.66 and 86.60°, respectively, compared to 88.71 (9)° in (I) and 84.59 (8)° in (II).

A search for partial structure *S*2 (Fig. 6) gave 23 hits. Details are given in in the supporting information (CSD search S2). In these structures, the dihedral angle between the planar pyrrolidine ring of the indole ring system and the mean plane of the pyrrolidine ring of the pyrrolothiazole unit varies from 77.60° in 1′-phenyl-6′-thiacycloheptane-1-spiro-2′-perhydro-pyrrolizine-3′-spiro-3″-indoline-2,2″-dione (GITDOD; Sundaramoorthy *et al.*, 2008) to 89.72° in 3-hydroxy-10,13-dimethyl-7′-(4-methylphenyl)-1,3,4,5,6,7,7′,7a′,8,9,10,11,12,-13,14,15-hexadecahydro-1′*H*-dispiro[cyclopenta[*a*]phenanthrene-16,6′-pyrrolo[1,2-*c*][1,3]thiazole-5′,3″-indole]-2″,17-(1″*H*,2*H*)-dione (MUDLAA; Kanchithalaivan *et al.*, 2014*b*). Only four of these compounds are monospiro, the others, like the two above, have a dispiro arrangement. The four compounds are 7′-(2-chlorophenyl)-6′-(pyridin-2-ylcarbonyl)-1′,6′,7′,7a′-tetrahydrospiro[indole-3,5′-pyrrolo[1,2-*c*][1,3]thiazol]-2(*1H*)-one ethanol solvate (GUCHET; Li *et al.*, 2014), ethyl 7′-(6-(benzyloxy)-2,2-dimethyltetrahydrofuro[2,3-*d*]-[1,3]dioxol-5-yl)-2-oxo-1,1′,2,6′,7′,7a′-hexahydrospiro[indole-3,5′-pyrrolo[1,2-*c*][1,3]thiazole]-6′-carboxylate (NUHHIJ; Suhitha *et al.*, 2013), ethyl 2-oxo-7′-(2,2,7,7-tetramethyltetrahydro-3a*H*-bis[1,3]dioxolo[4,5-*b*:4′,5′-*d*]pyran-5-yl)-1,1′,2,6′,-7′,7a′-hexahydrospiro[indole-3,5′-pyrrolo[1,2-*c*][1,3]thiazole]-6′-carboxylate monohydrate (SUWNEE; Prasanna *et al.*, 2010) and 6′-benzoyl-7′-(4-chlorophenyl)-3′-phenyl-1′,6′,7′,7a′-tetrahydrospiro[indole-3,5′-pyrrolo[1,2-*c*][1,3]thiazol]-2(1*H*)-one (XEVGIQ; Kumar *et al.*, 2013). Here the dihedral angles between the planar pyrrolidine ring of the indole ring system and the mean plane of the pyrrolidine ring of the pyrrolo-thiazole unit are 79.94, 87.79, 84.78 and 81.44°, respectively, compared to 88.71 (9)° in (I) and 84.59 (8)° in (II).

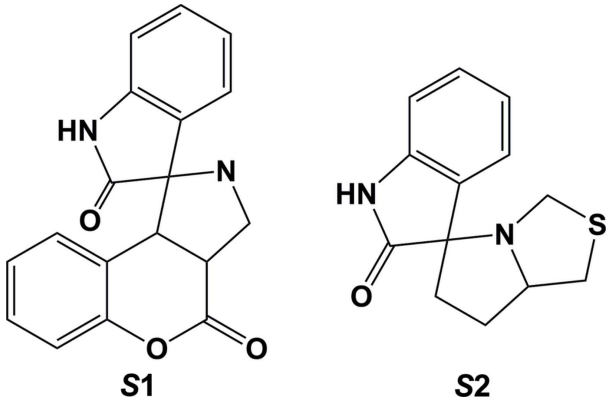



**Figure 6**
Partial structures for the CSD database searches.

## 5. Synthesis and crystallization

Compound (I): A flask containing salicylaldehyde (1 mmol) and 2,2-dimethyl-1,3-dioxane-4,6-dione (1 mmol) in water (7 ml) was placed at the maximum energy area in an ultrasonic cleaner and the surface of the reactants was placed slightly lower than the level of the water. The mixture was subjected to ultrasonic irradiation of low power at 323 K for *ca* 30 min. To this, a mixture of isatin (1 mmol) and 1,3-thiazolane-4-carboxylic acid (1 mmol) dissolved in methanol (7 ml) was added. The irradiation was continued until the completion of the reaction (*ca* 50 min), during which time the product precipitated from the reaction mixture. It was then filtered and dried to obtain the pure product. The compound was further recrystallized from an ethanol–ethyl acetate mixture (1:1) to obtain colourless block-like crystals.

Compound (II): A flask containing salicylaldehyde (1 mmol) and 2,2-dimethyl-1,3-dioxane-4,6-dione (1 mmol) in water (7 ml) was placed at the maximum energy area in an ultrasonic cleaner and the surface of the reactants was placed slightly lower than the level of the water. The mixture was subjected to ultrasonic irradiation of low power at 323 K for about 30 min. To this, a mixture of 5-methylisatin (1 mmol) and 1,3-thiazolane-4-carboxylic acid (1 mmol) dissolved in methanol (7 ml) was added. The irradiation was continued until the completion of the reaction (*ca* 45 min), during which time the product precipitated from the reaction mixture. It was then filtered and dried to obtain the pure product. The compound was further recrystallized from ethyl acetate to obtain colourless block-like crystals.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. For both compounds, the H atoms were placed in idealized positions and allowed to ride on their parent atoms: N—H = 0.86 Å and C—H = 0.93–0.97 Å, with $U_{iso}$(H) = 1.5$U_{eq}$(C-methyl) and 1.2$U_{eq}$(N, C) for other H atoms.

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**Table 3**
Experimental details.

| | (I) | (II) |
|---|---|---|
| Crystal data | | |
| Chemical formula | $C_{20}H_{16}N_2O_3S$ | $C_{21}H_{18}N_2O_3S$ |
| $M_r$ | 364.41 | 378.43 |
| Crystal system, space group | Monoclinic, $P2_1/n$ | Triclinic, $P\overline{1}$ |
| Temperature (K) | 298 | 298 |
| $a, b, c$ (Å) | 11.3058 (9), 10.0905 (8), 15.1957 (12) | 8.3648 (5), 9.7648 (6), 11.9677 (7) |
| $\alpha, \beta, \gamma$ (°) | 90, 101.072 (1), 90 | 112.622 (1), 99.388 (1), 91.885 (1) |
| $V$ (Å$^3$) | 1701.3 (2) | 885.31 (9) |
| $Z$ | 4 | 2 |
| Radiation type | Mo $K\alpha$ | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.21 | 0.21 |
| Crystal size (mm) | 0.21 × 0.18 × 0.16 | 0.22 × 0.19 × 0.17 |
| Data collection | | |
| Diffractometer | Bruker SMART APEX CCD area-detector | Bruker SMART APEX CCD area-detector |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 19453, 4146, 3646 | 10444, 4164, 3747 |
| $R_{int}$ | 0.023 | 0.016 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.668 | 0.666 |
| Refinement | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.052, 0.141, 1.02 | 0.048, 0.140, 1.05 |
| No. of reflections | 4146 | 4164 |
| No. of parameters | 235 | 245 |
| No. of restraints | 1 | 0 |
| H-atom treatment | H-atom parameters constrained | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.65, −0.37 | 0.67, −0.58 |

Computer programs: *SMART* and *SAINT* (Bruker, 2002), *SHELXS97* (Sheldrick, 2008), *SHELXL2018* (Sheldrick, 2015), *PLATON* (Spek, 2009), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).


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# supporting information

*Acta Cryst.* (2019). E**75**, 246-250 [https://doi.org/10.1107/S2056989019000045]

# Crystal structures of 6a,6b,7,11a-tetrahydro-6*H*,9*H*-spiro-[chromeno[3′,4′:3,4]pyrrolo[1,2-*c*]thiazole-11,3′-indoline]-2′,6-dione and 5′-methyl-6a,6b,7,11a-tetrahydro-6*H*,9*H*-spiro[chromeno[3′,4′:3,4]pyrrolo[1,2-*c*]thiazole-11,3′-indoline]-2′,6-dione

**S. Pangajavalli, R. Ranjithkumar, N. Srinivasan, S. Ramaswamy and S. Selvanayagam**

## Computing details

For both structures, data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT* (Bruker, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2018* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

### 6a,6b,7,11a-Tetrahydro-6*H*,9*H*-spiro[chromeno[3′,4′:3,4]pyrrolo[1,2-*c*]thiazole-11,3′-indoline]-2′,6-dione (I)

*Crystal data*

$C_{20}H_{16}N_2O_3S$
$M_r$ = 364.41
Monoclinic, $P2_1/n$
$a$ = 11.3058 (9) Å
$b$ = 10.0905 (8) Å
$c$ = 15.1957 (12) Å
$\beta$ = 101.072 (1)°
$V$ = 1701.3 (2) Å$^3$
$Z$ = 4
$F(000)$ = 760
$D_x$ = 1.423 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 11828 reflections
$\theta$ = 2.4–27.6°
$\mu$ = 0.21 mm$^{-1}$
$T$ = 298 K
Block, colourless
0.21 × 0.18 × 0.16 mm

*Data collection*

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
$\omega$ and $\varphi$ scans
19453 measured reflections
4146 independent reflections
3646 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.023
$\theta_{max}$ = 28.4°, $\theta_{min}$ = 2.1°
$h$ = −15→15
$k$ = −13→13
$l$ = −19→20

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.141
$S$ = 1.02
4146 reflections
235 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0749P)^2 + 0.6691P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.65$ e Å$^{-3}$
$\Delta\rho_{min} = -0.37$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.57371 (5) | 0.86992 (6) | 0.95071 (3) | 0.06229 (18) |
| O1 | 0.84530 (11) | 0.85243 (14) | 0.62287 (8) | 0.0483 (3) |
| O2 | 0.91582 (12) | 0.96789 (15) | 0.74380 (10) | 0.0580 (4) |
| O3 | 0.63036 (12) | 1.05167 (12) | 0.61274 (9) | 0.0491 (3) |
| N1 | 0.61304 (11) | 0.95792 (13) | 0.79329 (8) | 0.0363 (3) |
| N2 | 0.42770 (14) | 1.00915 (16) | 0.59558 (11) | 0.0510 (4) |
| H2 | 0.401330 | 1.064837 | 0.553539 | 0.061* |
| C1 | 0.84352 (14) | 0.88731 (17) | 0.70943 (12) | 0.0414 (4) |
| C2 | 0.75317 (15) | 0.77756 (17) | 0.57221 (11) | 0.0413 (4) |
| C3 | 0.77089 (19) | 0.7398 (2) | 0.48794 (13) | 0.0545 (5) |
| H3 | 0.842381 | 0.760585 | 0.469330 | 0.065* |
| C4 | 0.6811 (2) | 0.6711 (2) | 0.43215 (13) | 0.0619 (5) |
| H4 | 0.692571 | 0.644363 | 0.375835 | 0.074* |
| C5 | 0.5742 (2) | 0.6417 (2) | 0.45907 (13) | 0.0585 (5) |
| H5 | 0.512648 | 0.598674 | 0.420168 | 0.070* |
| C6 | 0.55927 (18) | 0.67675 (18) | 0.54432 (13) | 0.0496 (4) |
| H6 | 0.487889 | 0.655046 | 0.562774 | 0.060* |
| C7 | 0.64923 (15) | 0.74391 (15) | 0.60294 (11) | 0.0383 (3) |
| C8 | 0.63887 (13) | 0.77552 (15) | 0.69741 (10) | 0.0351 (3) |
| H8 | 0.608885 | 0.695840 | 0.722794 | 0.042* |
| C9 | 0.75974 (14) | 0.81137 (16) | 0.75678 (10) | 0.0382 (3) |
| H9 | 0.799982 | 0.729271 | 0.780519 | 0.046* |
| C10 | 0.72344 (14) | 0.88742 (18) | 0.83418 (10) | 0.0417 (4) |
| H10 | 0.786632 | 0.950538 | 0.859742 | 0.050* |
| C11 | 0.6941 (2) | 0.7942 (2) | 0.90799 (13) | 0.0611 (6) |
| H11A | 0.764283 | 0.783469 | 0.955452 | 0.073* |
| H11B | 0.669905 | 0.707638 | 0.883100 | 0.073* |
| C12 | 0.54452 (15) | 0.99367 (18) | 0.86045 (11) | 0.0433 (4) |
| H12A | 0.459238 | 0.995734 | 0.834284 | 0.052* |
| H12B | 0.568283 | 1.080964 | 0.884175 | 0.052* |
| C13 | 0.55309 (13) | 0.89158 (14) | 0.70978 (10) | 0.0325 (3) |
| C14 | 0.54547 (15) | 0.99491 (15) | 0.63379 (10) | 0.0375 (3) |
| C15 | 0.35371 (15) | 0.92197 (19) | 0.63287 (12) | 0.0465 (4) |
| C16 | 0.23026 (18) | 0.9034 (3) | 0.60850 (16) | 0.0679 (6) |
| H16 | 0.183914 | 0.953834 | 0.563437 | 0.081* |
| C17 | 0.17892 (19) | 0.8076 (3) | 0.65336 (18) | 0.0767 (8) |

| | | | | |
|---|---|---|---|---|
| H17 | 0.096136 | 0.793850 | 0.638706 | 0.092* |
| C18 | 0.2469 (2) | 0.7318 (3) | 0.71934 (17) | 0.0718 (7) |
| H18 | 0.209658 | 0.666702 | 0.747758 | 0.086* |
| C19 | 0.37089 (18) | 0.7509 (2) | 0.74433 (13) | 0.0547 (5) |
| H19 | 0.417048 | 0.699062 | 0.788679 | 0.066* |
| C20 | 0.42334 (14) | 0.84931 (17) | 0.70121 (10) | 0.0391 (3) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0646 (3) | 0.0793 (4) | 0.0497 (3) | 0.0140 (3) | 0.0277 (2) | 0.0133 (2) |
| O1 | 0.0390 (6) | 0.0624 (8) | 0.0475 (7) | 0.0000 (5) | 0.0180 (5) | 0.0005 (6) |
| O2 | 0.0435 (7) | 0.0635 (8) | 0.0679 (9) | −0.0092 (6) | 0.0132 (6) | −0.0084 (7) |
| O3 | 0.0530 (7) | 0.0456 (7) | 0.0512 (7) | −0.0028 (5) | 0.0164 (5) | 0.0105 (5) |
| N1 | 0.0341 (6) | 0.0409 (7) | 0.0333 (6) | 0.0016 (5) | 0.0047 (5) | −0.0035 (5) |
| N2 | 0.0463 (8) | 0.0557 (9) | 0.0473 (8) | 0.0107 (7) | −0.0002 (6) | 0.0104 (7) |
| C1 | 0.0321 (7) | 0.0452 (9) | 0.0481 (9) | 0.0056 (6) | 0.0107 (6) | 0.0017 (7) |
| C2 | 0.0430 (8) | 0.0418 (8) | 0.0405 (8) | 0.0097 (7) | 0.0115 (7) | 0.0029 (6) |
| C3 | 0.0605 (11) | 0.0619 (11) | 0.0458 (9) | 0.0179 (9) | 0.0218 (8) | 0.0045 (8) |
| C4 | 0.0869 (15) | 0.0603 (12) | 0.0398 (9) | 0.0177 (11) | 0.0152 (9) | −0.0056 (8) |
| C5 | 0.0756 (14) | 0.0496 (10) | 0.0463 (10) | 0.0016 (9) | 0.0017 (9) | −0.0086 (8) |
| C6 | 0.0549 (10) | 0.0423 (9) | 0.0512 (10) | −0.0011 (8) | 0.0090 (8) | −0.0056 (7) |
| C7 | 0.0443 (8) | 0.0322 (7) | 0.0394 (8) | 0.0060 (6) | 0.0105 (6) | −0.0008 (6) |
| C8 | 0.0366 (7) | 0.0314 (7) | 0.0388 (7) | 0.0015 (6) | 0.0111 (6) | 0.0026 (6) |
| C9 | 0.0357 (7) | 0.0418 (8) | 0.0376 (7) | 0.0066 (6) | 0.0081 (6) | 0.0046 (6) |
| C10 | 0.0334 (7) | 0.0560 (10) | 0.0352 (8) | 0.0038 (7) | 0.0053 (6) | −0.0002 (7) |
| C11 | 0.0646 (12) | 0.0792 (14) | 0.0438 (9) | 0.0259 (11) | 0.0210 (9) | 0.0182 (9) |
| C12 | 0.0431 (8) | 0.0475 (9) | 0.0399 (8) | 0.0052 (7) | 0.0090 (6) | −0.0078 (7) |
| C13 | 0.0313 (7) | 0.0341 (7) | 0.0328 (7) | −0.0005 (5) | 0.0079 (5) | −0.0005 (5) |
| C14 | 0.0419 (8) | 0.0348 (7) | 0.0362 (7) | 0.0040 (6) | 0.0087 (6) | −0.0006 (6) |
| C15 | 0.0356 (8) | 0.0571 (10) | 0.0457 (9) | 0.0043 (7) | 0.0054 (7) | −0.0124 (8) |
| C16 | 0.0381 (10) | 0.0904 (16) | 0.0697 (13) | 0.0066 (10) | −0.0029 (9) | −0.0207 (12) |
| C17 | 0.0366 (10) | 0.111 (2) | 0.0841 (16) | −0.0196 (12) | 0.0150 (10) | −0.0381 (15) |
| C18 | 0.0551 (12) | 0.0933 (17) | 0.0739 (14) | −0.0346 (12) | 0.0293 (11) | −0.0258 (13) |
| C19 | 0.0510 (10) | 0.0648 (12) | 0.0519 (10) | −0.0176 (9) | 0.0192 (8) | −0.0071 (9) |
| C20 | 0.0332 (7) | 0.0479 (9) | 0.0379 (8) | −0.0037 (6) | 0.0108 (6) | −0.0097 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C11 | 1.789 (2) | C8—C9 | 1.529 (2) |
| S1—C12 | 1.8373 (19) | C8—C13 | 1.555 (2) |
| O1—C1 | 1.366 (2) | C8—H8 | 0.9800 |
| O1—C2 | 1.393 (2) | C9—C10 | 1.525 (2) |
| O2—C1 | 1.199 (2) | C9—H9 | 0.9800 |
| O3—C14 | 1.212 (2) | C10—C11 | 1.548 (3) |
| N1—C12 | 1.441 (2) | C10—H10 | 0.9800 |
| N1—C10 | 1.467 (2) | C11—H11A | 0.9700 |
| N1—C13 | 1.4791 (19) | C11—H11B | 0.9700 |

| | | | |
|---|---|---|---|
| N2—C14 | 1.354 (2) | C12—H12A | 0.9700 |
| N2—C15 | 1.406 (3) | C12—H12B | 0.9700 |
| N2—H2 | 0.8600 | C13—C20 | 1.509 (2) |
| C1—C9 | 1.505 (2) | C13—C14 | 1.546 (2) |
| C2—C3 | 1.387 (2) | C15—C20 | 1.386 (3) |
| C2—C7 | 1.387 (2) | C15—C16 | 1.386 (3) |
| C3—C4 | 1.378 (3) | C16—C17 | 1.374 (4) |
| C3—H3 | 0.9300 | C16—H16 | 0.9300 |
| C4—C5 | 1.380 (3) | C17—C18 | 1.373 (4) |
| C4—H4 | 0.9300 | C17—H17 | 0.9300 |
| C5—C6 | 1.384 (3) | C18—C19 | 1.394 (3) |
| C5—H5 | 0.9300 | C18—H18 | 0.9300 |
| C6—C7 | 1.393 (2) | C19—C20 | 1.384 (3) |
| C6—H6 | 0.9300 | C19—H19 | 0.9300 |
| C7—C8 | 1.497 (2) | | |

| | | | |
|---|---|---|---|
| C11—S1—C12 | 93.42 (8) | C9—C10—C11 | 112.32 (15) |
| C1—O1—C2 | 121.62 (13) | N1—C10—H10 | 110.4 |
| C12—N1—C10 | 110.55 (12) | C9—C10—H10 | 110.4 |
| C12—N1—C13 | 120.07 (13) | C11—C10—H10 | 110.4 |
| C10—N1—C13 | 110.87 (12) | C10—C11—S1 | 106.68 (13) |
| C14—N2—C15 | 111.89 (14) | C10—C11—H11A | 110.4 |
| C14—N2—H2 | 124.1 | S1—C11—H11A | 110.4 |
| C15—N2—H2 | 124.1 | C10—C11—H11B | 110.4 |
| O2—C1—O1 | 117.34 (16) | S1—C11—H11B | 110.4 |
| O2—C1—C9 | 125.18 (17) | H11A—C11—H11B | 108.6 |
| O1—C1—C9 | 117.17 (15) | N1—C12—S1 | 108.34 (11) |
| C3—C2—C7 | 121.70 (17) | N1—C12—H12A | 110.0 |
| C3—C2—O1 | 115.71 (16) | S1—C12—H12A | 110.0 |
| C7—C2—O1 | 122.58 (14) | N1—C12—H12B | 110.0 |
| C4—C3—C2 | 119.08 (19) | S1—C12—H12B | 110.0 |
| C4—C3—H3 | 120.5 | H12A—C12—H12B | 108.4 |
| C2—C3—H3 | 120.5 | N1—C13—C20 | 118.66 (12) |
| C3—C4—C5 | 120.61 (18) | N1—C13—C14 | 106.63 (12) |
| C3—C4—H4 | 119.7 | C20—C13—C14 | 102.26 (12) |
| C5—C4—H4 | 119.7 | N1—C13—C8 | 104.53 (11) |
| C4—C5—C6 | 119.59 (19) | C20—C13—C8 | 113.28 (12) |
| C4—C5—H5 | 120.2 | C14—C13—C8 | 111.37 (12) |
| C6—C5—H5 | 120.2 | O3—C14—N2 | 126.85 (16) |
| C5—C6—C7 | 121.16 (19) | O3—C14—C13 | 125.62 (14) |
| C5—C6—H6 | 119.4 | N2—C14—C13 | 107.52 (14) |
| C7—C6—H6 | 119.4 | C20—C15—C16 | 121.8 (2) |
| C2—C7—C6 | 117.74 (16) | C20—C15—N2 | 109.67 (14) |
| C2—C7—C8 | 119.86 (15) | C16—C15—N2 | 128.57 (19) |
| C6—C7—C8 | 122.36 (15) | C17—C16—C15 | 117.5 (2) |
| C7—C8—C9 | 112.99 (13) | C17—C16—H16 | 121.2 |
| C7—C8—C13 | 116.23 (12) | C15—C16—H16 | 121.2 |
| C9—C8—C13 | 104.95 (12) | C18—C17—C16 | 121.6 (2) |

| | | | |
|---|---|---|---|
| C7—C8—H8 | 107.4 | C18—C17—H17 | 119.2 |
| C9—C8—H8 | 107.4 | C16—C17—H17 | 119.2 |
| C13—C8—H8 | 107.4 | C17—C18—C19 | 121.0 (2) |
| C1—C9—C10 | 113.58 (14) | C17—C18—H18 | 119.5 |
| C1—C9—C8 | 114.30 (13) | C19—C18—H18 | 119.5 |
| C10—C9—C8 | 103.39 (12) | C20—C19—C18 | 118.0 (2) |
| C1—C9—H9 | 108.4 | C20—C19—H19 | 121.0 |
| C10—C9—H9 | 108.4 | C18—C19—H19 | 121.0 |
| C8—C9—H9 | 108.4 | C19—C20—C15 | 120.05 (16) |
| N1—C10—C9 | 104.51 (12) | C19—C20—C13 | 131.26 (16) |
| N1—C10—C11 | 108.58 (13) | C15—C20—C13 | 108.54 (14) |

| | | | |
|---|---|---|---|
| C2—O1—C1—O2 | −168.97 (15) | C11—S1—C12—N1 | 9.94 (14) |
| C2—O1—C1—C9 | 17.2 (2) | C12—N1—C13—C20 | −6.6 (2) |
| C1—O1—C2—C3 | −174.90 (15) | C10—N1—C13—C20 | 124.34 (15) |
| C1—O1—C2—C7 | 6.0 (2) | C12—N1—C13—C14 | 108.01 (15) |
| C7—C2—C3—C4 | 2.3 (3) | C10—N1—C13—C14 | −121.09 (13) |
| O1—C2—C3—C4 | −176.83 (16) | C12—N1—C13—C8 | −133.94 (14) |
| C2—C3—C4—C5 | 0.9 (3) | C10—N1—C13—C8 | −3.04 (15) |
| C3—C4—C5—C6 | −2.7 (3) | C7—C8—C13—N1 | −143.84 (13) |
| C4—C5—C6—C7 | 1.4 (3) | C9—C8—C13—N1 | −18.23 (15) |
| C3—C2—C7—C6 | −3.5 (3) | C7—C8—C13—C20 | 85.55 (16) |
| O1—C2—C7—C6 | 175.57 (15) | C9—C8—C13—C20 | −148.84 (12) |
| C3—C2—C7—C8 | 174.22 (15) | C7—C8—C13—C14 | −29.07 (18) |
| O1—C2—C7—C8 | −6.7 (2) | C9—C8—C13—C14 | 96.54 (14) |
| C5—C6—C7—C2 | 1.6 (3) | C15—N2—C14—O3 | 176.23 (16) |
| C5—C6—C7—C8 | −176.06 (17) | C15—N2—C14—C13 | −3.47 (19) |
| C2—C7—C8—C9 | −14.6 (2) | N1—C13—C14—O3 | 58.27 (19) |
| C6—C7—C8—C9 | 162.98 (15) | C20—C13—C14—O3 | −176.48 (15) |
| C2—C7—C8—C13 | 106.78 (17) | C8—C13—C14—O3 | −55.2 (2) |
| C6—C7—C8—C13 | −75.59 (19) | N1—C13—C14—N2 | −122.03 (14) |
| O2—C1—C9—C10 | 30.5 (2) | C20—C13—C14—N2 | 3.23 (16) |
| O1—C1—C9—C10 | −156.16 (14) | C8—C13—C14—N2 | 124.51 (14) |
| O2—C1—C9—C8 | 148.82 (17) | C14—N2—C15—C20 | 2.3 (2) |
| O1—C1—C9—C8 | −37.8 (2) | C14—N2—C15—C16 | −176.71 (19) |
| C7—C8—C9—C1 | 35.47 (18) | C20—C15—C16—C17 | −1.4 (3) |
| C13—C8—C9—C1 | −92.13 (15) | N2—C15—C16—C17 | 177.50 (19) |
| C7—C8—C9—C10 | 159.44 (13) | C15—C16—C17—C18 | −0.7 (3) |
| C13—C8—C9—C10 | 31.84 (15) | C16—C17—C18—C19 | 1.1 (4) |
| C12—N1—C10—C9 | 158.83 (13) | C17—C18—C19—C20 | 0.5 (3) |
| C13—N1—C10—C9 | 23.14 (17) | C18—C19—C20—C15 | −2.5 (3) |
| C12—N1—C10—C11 | 38.78 (19) | C18—C19—C20—C13 | −177.54 (17) |
| C13—N1—C10—C11 | −96.91 (16) | C16—C15—C20—C19 | 3.0 (3) |
| C1—C9—C10—N1 | 90.83 (16) | N2—C15—C20—C19 | −176.04 (16) |
| C8—C9—C10—N1 | −33.61 (16) | C16—C15—C20—C13 | 179.08 (17) |
| C1—C9—C10—C11 | −151.65 (15) | N2—C15—C20—C13 | 0.01 (18) |
| C8—C9—C10—C11 | 83.91 (16) | N1—C13—C20—C19 | −69.5 (2) |
| N1—C10—C11—S1 | −29.53 (19) | C14—C13—C20—C19 | 173.54 (17) |

| | | | |
|---|---|---|---|
| C9—C10—C11—S1 | −144.59 (13) | C8—C13—C20—C19 | 53.6 (2) |
| C12—S1—C11—C10 | 11.05 (16) | N1—C13—C20—C15 | 115.00 (15) |
| C10—N1—C12—S1 | −29.65 (16) | C14—C13—C20—C15 | −1.91 (16) |
| C13—N1—C12—S1 | 101.39 (14) | C8—C13—C20—C15 | −121.87 (14) |

*Hydrogen-bond geometry (Å, º)*

Cg is the centroid of the C2–C7 ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C10—H10···O2 | 0.98 | 2.50 | 2.902 (2) | 104 |
| N2—H2···*Cg*$^{i}$ | 0.86 | 2.57 | 3.799 (18) | 157 |
| C8—H8···O2$^{ii}$ | 0.98 | 2.38 | 3.321 (2) | 160 |
| C9—H9···O3$^{ii}$ | 0.98 | 2.44 | 3.376 (2) | 159 |

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

## 5′-Methyl-6a,6b,7,11a-tetrahydro-6*H*,9*H*-spiro[chromeno[3′,4′:3,4]pyrrolo[1,2-*c*]thiazole-11,3′-indoline]-2′,6-dione (II)

*Crystal data*

$C_{21}H_{18}N_2O_3S$
$M_r$ = 378.43
Triclinic, $P\overline{1}$
*a* = 8.3648 (5) Å
*b* = 9.7648 (6) Å
*c* = 11.9677 (7) Å
*α* = 112.622 (1)°
*β* = 99.388 (1)°
*γ* = 91.885 (1)°
*V* = 885.31 (9) Å$^3$
*Z* = 2
*F*(000) = 396
$D_x$ = 1.420 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 7888 reflections
*θ* = 1.9–27.2°
*μ* = 0.21 mm$^{-1}$
*T* = 298 K
Block, colourless
0.22 × 0.19 × 0.17 mm

*Data collection*

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
*ω* and *φ* scans
10444 measured reflections
4164 independent reflections
3747 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.016
$\theta_{max}$ = 28.3°, $\theta_{min}$ = 1.9°
*h* = −11→10
*k* = −12→12
*l* = −15→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.140
*S* = 1.05
4164 reflections
245 parameters
0 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0815P)^2 + 0.2689P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.67 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.58 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | −0.29132 (5) | 0.74000 (6) | 0.23215 (5) | 0.06325 (18) |
| O1 | 0.28203 (16) | 0.52624 (15) | 0.43732 (11) | 0.0523 (3) |
| O2 | 0.13654 (18) | 0.64554 (16) | 0.57307 (11) | 0.0577 (3) |
| O3 | 0.33381 (14) | 0.85985 (13) | 0.48566 (10) | 0.0430 (3) |
| N1 | 0.00582 (14) | 0.86415 (13) | 0.35425 (11) | 0.0349 (3) |
| N2 | 0.38289 (15) | 0.95200 (14) | 0.34299 (12) | 0.0385 (3) |
| H2 | 0.470194 | 1.010156 | 0.384421 | 0.046* |
| C1 | 0.1543 (2) | 0.60502 (18) | 0.46820 (14) | 0.0429 (3) |
| C2 | 0.3272 (2) | 0.48986 (17) | 0.32294 (14) | 0.0412 (3) |
| C3 | 0.4559 (2) | 0.4032 (2) | 0.30405 (18) | 0.0530 (4) |
| H3 | 0.504909 | 0.372078 | 0.364539 | 0.064* |
| C4 | 0.5103 (2) | 0.3637 (2) | 0.19411 (19) | 0.0543 (4) |
| H4 | 0.597736 | 0.306867 | 0.180813 | 0.065* |
| C5 | 0.4360 (2) | 0.4081 (2) | 0.10393 (18) | 0.0516 (4) |
| H5 | 0.472276 | 0.380135 | 0.029583 | 0.062* |
| C6 | 0.3071 (2) | 0.49431 (17) | 0.12417 (15) | 0.0415 (3) |
| H6 | 0.257290 | 0.523540 | 0.062749 | 0.050* |
| C7 | 0.25060 (17) | 0.53824 (15) | 0.23529 (13) | 0.0344 (3) |
| C8 | 0.10802 (17) | 0.62702 (14) | 0.25841 (12) | 0.0304 (3) |
| H8 | 0.022930 | 0.584116 | 0.184089 | 0.037* |
| C9 | 0.03505 (18) | 0.62333 (16) | 0.36667 (12) | 0.0342 (3) |
| H9 | −0.050188 | 0.539144 | 0.334102 | 0.041* |
| C10 | −0.04772 (19) | 0.76820 (17) | 0.41498 (13) | 0.0384 (3) |
| H10 | −0.011738 | 0.818793 | 0.504508 | 0.046* |
| C11 | −0.2345 (2) | 0.7416 (2) | 0.3835 (2) | 0.0562 (5) |
| H11A | −0.281609 | 0.820745 | 0.442410 | 0.067* |
| H11B | −0.271795 | 0.647102 | 0.384579 | 0.067* |
| C12 | −0.1351 (2) | 0.8969 (2) | 0.28430 (17) | 0.0490 (4) |
| H12A | −0.105957 | 0.911235 | 0.214080 | 0.059* |
| H12B | −0.175301 | 0.987402 | 0.335626 | 0.059* |
| C13 | 0.13847 (16) | 0.79580 (15) | 0.28968 (12) | 0.0310 (3) |
| C14 | 0.29735 (17) | 0.87016 (15) | 0.38586 (13) | 0.0338 (3) |
| C15 | 0.31155 (17) | 0.93035 (16) | 0.22215 (14) | 0.0355 (3) |
| C16 | 0.3694 (2) | 0.98465 (19) | 0.14429 (16) | 0.0463 (4) |
| H16 | 0.466289 | 1.047091 | 0.169880 | 0.056* |
| C17 | 0.2784 (2) | 0.94312 (19) | 0.02629 (16) | 0.0470 (4) |
| H17 | 0.315011 | 0.980334 | −0.026878 | 0.056* |
| C18 | 0.1343 (2) | 0.84777 (16) | −0.01552 (14) | 0.0398 (3) |
| C19 | 0.07923 (19) | 0.79402 (15) | 0.06572 (13) | 0.0365 (3) |

| | | | | |
|---|---|---|---|---|
| H19 | −0.015862 | 0.729181 | 0.039691 | 0.044* |
| C20 | 0.16607 (17) | 0.83718 (14) | 0.18489 (13) | 0.0321 (3) |
| C21 | 0.0385 (3) | 0.8044 (2) | −0.14405 (15) | 0.0522 (4) |
| H21A | −0.023462 | 0.884117 | −0.147536 | 0.078* |
| H21B | −0.034059 | 0.716052 | −0.165745 | 0.078* |
| H21C | 0.111978 | 0.785222 | −0.200908 | 0.078* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0352 (2) | 0.0722 (3) | 0.0593 (3) | 0.0004 (2) | −0.00072 (19) | 0.0054 (2) |
| O1 | 0.0604 (8) | 0.0633 (8) | 0.0366 (6) | 0.0138 (6) | 0.0050 (5) | 0.0245 (6) |
| O2 | 0.0719 (9) | 0.0725 (9) | 0.0334 (6) | 0.0018 (7) | 0.0122 (6) | 0.0255 (6) |
| O3 | 0.0408 (6) | 0.0495 (6) | 0.0329 (5) | −0.0105 (5) | −0.0009 (4) | 0.0144 (5) |
| N1 | 0.0321 (6) | 0.0334 (6) | 0.0370 (6) | −0.0014 (4) | 0.0093 (5) | 0.0109 (5) |
| N2 | 0.0311 (6) | 0.0413 (7) | 0.0378 (6) | −0.0093 (5) | 0.0043 (5) | 0.0120 (5) |
| C1 | 0.0512 (9) | 0.0462 (8) | 0.0328 (7) | −0.0035 (7) | 0.0057 (6) | 0.0188 (6) |
| C2 | 0.0433 (8) | 0.0394 (7) | 0.0361 (7) | 0.0003 (6) | 0.0026 (6) | 0.0120 (6) |
| C3 | 0.0510 (10) | 0.0467 (9) | 0.0545 (10) | 0.0085 (7) | −0.0017 (8) | 0.0171 (8) |
| C4 | 0.0463 (9) | 0.0441 (9) | 0.0646 (11) | 0.0098 (7) | 0.0112 (8) | 0.0124 (8) |
| C5 | 0.0528 (10) | 0.0450 (9) | 0.0531 (10) | 0.0035 (7) | 0.0211 (8) | 0.0110 (7) |
| C6 | 0.0462 (8) | 0.0383 (7) | 0.0386 (8) | 0.0020 (6) | 0.0115 (6) | 0.0126 (6) |
| C7 | 0.0361 (7) | 0.0298 (6) | 0.0330 (7) | −0.0029 (5) | 0.0047 (5) | 0.0091 (5) |
| C8 | 0.0335 (6) | 0.0303 (6) | 0.0254 (6) | −0.0037 (5) | 0.0041 (5) | 0.0100 (5) |
| C9 | 0.0385 (7) | 0.0351 (7) | 0.0280 (6) | −0.0053 (5) | 0.0070 (5) | 0.0118 (5) |
| C10 | 0.0426 (8) | 0.0384 (7) | 0.0312 (7) | −0.0027 (6) | 0.0121 (6) | 0.0093 (6) |
| C11 | 0.0434 (9) | 0.0535 (10) | 0.0752 (13) | 0.0006 (7) | 0.0276 (9) | 0.0233 (9) |
| C12 | 0.0419 (8) | 0.0568 (10) | 0.0570 (10) | 0.0120 (7) | 0.0164 (7) | 0.0286 (8) |
| C13 | 0.0302 (6) | 0.0305 (6) | 0.0295 (6) | −0.0040 (5) | 0.0048 (5) | 0.0097 (5) |
| C14 | 0.0306 (6) | 0.0315 (6) | 0.0338 (7) | −0.0028 (5) | 0.0062 (5) | 0.0073 (5) |
| C15 | 0.0343 (7) | 0.0340 (7) | 0.0376 (7) | −0.0003 (5) | 0.0100 (6) | 0.0127 (6) |
| C16 | 0.0438 (8) | 0.0475 (9) | 0.0511 (9) | −0.0062 (7) | 0.0158 (7) | 0.0214 (7) |
| C17 | 0.0567 (10) | 0.0471 (9) | 0.0470 (9) | 0.0038 (7) | 0.0235 (8) | 0.0240 (7) |
| C18 | 0.0538 (9) | 0.0330 (7) | 0.0344 (7) | 0.0076 (6) | 0.0137 (6) | 0.0132 (6) |
| C19 | 0.0425 (8) | 0.0311 (7) | 0.0342 (7) | −0.0015 (5) | 0.0061 (6) | 0.0118 (5) |
| C20 | 0.0338 (7) | 0.0289 (6) | 0.0335 (7) | −0.0006 (5) | 0.0081 (5) | 0.0117 (5) |
| C21 | 0.0774 (12) | 0.0452 (9) | 0.0342 (8) | 0.0052 (8) | 0.0107 (8) | 0.0159 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C11 | 1.790 (2) | C8—C13 | 1.5453 (18) |
| S1—C12 | 1.8191 (19) | C8—H8 | 0.9800 |
| O1—C1 | 1.355 (2) | C9—C10 | 1.543 (2) |
| O1—C2 | 1.395 (2) | C9—H9 | 0.9800 |
| O2—C1 | 1.1985 (19) | C10—C11 | 1.535 (2) |
| O3—C14 | 1.2270 (18) | C10—H10 | 0.9800 |
| N1—C12 | 1.450 (2) | C11—H11A | 0.9700 |
| N1—C13 | 1.4851 (17) | C11—H11B | 0.9700 |

| | | | |
|---|---|---|---|
| N1—C10 | 1.4862 (18) | C12—H12A | 0.9700 |
| N2—C14 | 1.3458 (18) | C12—H12B | 0.9700 |
| N2—C15 | 1.4026 (19) | C13—C20 | 1.5058 (18) |
| N2—H2 | 0.8600 | C13—C14 | 1.5534 (18) |
| C1—C9 | 1.512 (2) | C15—C16 | 1.376 (2) |
| C2—C7 | 1.384 (2) | C15—C20 | 1.3948 (19) |
| C2—C3 | 1.385 (2) | C16—C17 | 1.388 (2) |
| C3—C4 | 1.380 (3) | C16—H16 | 0.9300 |
| C3—H3 | 0.9300 | C17—C18 | 1.393 (2) |
| C4—C5 | 1.377 (3) | C17—H17 | 0.9300 |
| C4—H4 | 0.9300 | C18—C19 | 1.398 (2) |
| C5—C6 | 1.385 (2) | C18—C21 | 1.504 (2) |
| C5—H5 | 0.9300 | C19—C20 | 1.385 (2) |
| C6—C7 | 1.399 (2) | C19—H19 | 0.9300 |
| C6—H6 | 0.9300 | C21—H21A | 0.9600 |
| C7—C8 | 1.497 (2) | C21—H21B | 0.9600 |
| C8—C9 | 1.5314 (18) | C21—H21C | 0.9600 |

| | | | |
|---|---|---|---|
| C11—S1—C12 | 85.68 (8) | C9—C10—H10 | 109.6 |
| C1—O1—C2 | 122.33 (12) | C10—C11—S1 | 105.85 (11) |
| C12—N1—C13 | 118.56 (12) | C10—C11—H11A | 110.6 |
| C12—N1—C10 | 109.63 (12) | S1—C11—H11A | 110.6 |
| C13—N1—C10 | 108.19 (11) | C10—C11—H11B | 110.6 |
| C14—N2—C15 | 111.38 (11) | S1—C11—H11B | 110.6 |
| C14—N2—H2 | 124.3 | H11A—C11—H11B | 108.7 |
| C15—N2—H2 | 124.3 | N1—C12—S1 | 107.95 (11) |
| O2—C1—O1 | 117.29 (15) | N1—C12—H12A | 110.1 |
| O2—C1—C9 | 124.23 (17) | S1—C12—H12A | 110.1 |
| O1—C1—C9 | 118.21 (13) | N1—C12—H12B | 110.1 |
| C7—C2—C3 | 122.38 (16) | S1—C12—H12B | 110.1 |
| C7—C2—O1 | 122.58 (15) | H12A—C12—H12B | 108.4 |
| C3—C2—O1 | 115.03 (15) | N1—C13—C20 | 116.57 (11) |
| C4—C3—C2 | 118.98 (17) | N1—C13—C8 | 105.43 (10) |
| C4—C3—H3 | 120.5 | C20—C13—C8 | 115.84 (11) |
| C2—C3—H3 | 120.5 | N1—C13—C14 | 104.40 (10) |
| C5—C4—C3 | 120.40 (17) | C20—C13—C14 | 101.50 (11) |
| C5—C4—H4 | 119.8 | C8—C13—C14 | 112.66 (11) |
| C3—C4—H4 | 119.8 | O3—C14—N2 | 126.47 (13) |
| C4—C5—C6 | 119.87 (17) | O3—C14—C13 | 125.21 (12) |
| C4—C5—H5 | 120.1 | N2—C14—C13 | 108.26 (12) |
| C6—C5—H5 | 120.1 | C16—C15—C20 | 121.57 (14) |
| C5—C6—C7 | 121.20 (16) | C16—C15—N2 | 128.55 (14) |
| C5—C6—H6 | 119.4 | C20—C15—N2 | 109.85 (12) |
| C7—C6—H6 | 119.4 | C15—C16—C17 | 117.78 (15) |
| C2—C7—C6 | 117.15 (14) | C15—C16—H16 | 121.1 |
| C2—C7—C8 | 120.27 (13) | C17—C16—H16 | 121.1 |
| C6—C7—C8 | 122.51 (13) | C16—C17—C18 | 122.49 (14) |
| C7—C8—C9 | 113.73 (11) | C16—C17—H17 | 118.8 |

| | | | |
|---|---|---|---|
| C7—C8—C13 | 116.91 (11) | C18—C17—H17 | 118.8 |
| C9—C8—C13 | 102.92 (11) | C17—C18—C19 | 118.28 (14) |
| C7—C8—H8 | 107.6 | C17—C18—C21 | 121.29 (14) |
| C9—C8—H8 | 107.6 | C19—C18—C21 | 120.43 (15) |
| C13—C8—H8 | 107.6 | C20—C19—C18 | 120.12 (14) |
| C1—C9—C8 | 115.20 (12) | C20—C19—H19 | 119.9 |
| C1—C9—C10 | 111.88 (12) | C18—C19—H19 | 119.9 |
| C8—C9—C10 | 106.10 (11) | C19—C20—C15 | 119.71 (13) |
| C1—C9—H9 | 107.8 | C19—C20—C13 | 131.67 (12) |
| C8—C9—H9 | 107.8 | C15—C20—C13 | 108.54 (12) |
| C10—C9—H9 | 107.8 | C18—C21—H21A | 109.5 |
| N1—C10—C11 | 108.17 (13) | C18—C21—H21B | 109.5 |
| N1—C10—C9 | 106.68 (11) | H21A—C21—H21B | 109.5 |
| C11—C10—C9 | 113.15 (13) | C18—C21—H21C | 109.5 |
| N1—C10—H10 | 109.6 | H21A—C21—H21C | 109.5 |
| C11—C10—H10 | 109.6 | H21B—C21—H21C | 109.5 |

| | | | |
|---|---|---|---|
| C2—O1—C1—O2 | −171.58 (15) | C12—N1—C13—C20 | 31.56 (17) |
| C2—O1—C1—C9 | 14.1 (2) | C10—N1—C13—C20 | 157.10 (12) |
| C1—O1—C2—C7 | 3.6 (2) | C12—N1—C13—C8 | −98.51 (14) |
| C1—O1—C2—C3 | −177.22 (15) | C10—N1—C13—C8 | 27.03 (14) |
| C7—C2—C3—C4 | 0.2 (3) | C12—N1—C13—C14 | 142.58 (13) |
| O1—C2—C3—C4 | −179.05 (16) | C10—N1—C13—C14 | −91.88 (12) |
| C2—C3—C4—C5 | −1.0 (3) | C7—C8—C13—N1 | −158.43 (11) |
| C3—C4—C5—C6 | 0.8 (3) | C9—C8—C13—N1 | −33.02 (13) |
| C4—C5—C6—C7 | 0.2 (3) | C7—C8—C13—C20 | 71.07 (15) |
| C3—C2—C7—C6 | 0.8 (2) | C9—C8—C13—C20 | −163.51 (12) |
| O1—C2—C7—C6 | 179.97 (14) | C7—C8—C13—C14 | −45.18 (16) |
| C3—C2—C7—C8 | 177.75 (15) | C9—C8—C13—C14 | 80.23 (13) |
| O1—C2—C7—C8 | −3.1 (2) | C15—N2—C14—O3 | 176.06 (14) |
| C5—C6—C7—C2 | −1.0 (2) | C15—N2—C14—C13 | −6.65 (16) |
| C5—C6—C7—C8 | −177.86 (14) | N1—C13—C14—O3 | 62.34 (17) |
| C2—C7—C8—C9 | −14.12 (18) | C20—C13—C14—O3 | −176.09 (14) |
| C6—C7—C8—C9 | 162.65 (13) | C8—C13—C14—O3 | −51.55 (18) |
| C2—C7—C8—C13 | 105.69 (15) | N1—C13—C14—N2 | −115.00 (12) |
| C6—C7—C8—C13 | −77.55 (17) | C20—C13—C14—N2 | 6.58 (14) |
| O2—C1—C9—C8 | 155.28 (16) | C8—C13—C14—N2 | 131.12 (12) |
| O1—C1—C9—C8 | −30.88 (19) | C14—N2—C15—C16 | −174.38 (16) |
| O2—C1—C9—C10 | 34.0 (2) | C14—N2—C15—C20 | 3.89 (17) |
| O1—C1—C9—C10 | −152.16 (13) | C20—C15—C16—C17 | −0.3 (2) |
| C7—C8—C9—C1 | 29.87 (17) | N2—C15—C16—C17 | 177.81 (15) |
| C13—C8—C9—C1 | −97.58 (14) | C15—C16—C17—C18 | −1.1 (3) |
| C7—C8—C9—C10 | 154.24 (11) | C16—C17—C18—C19 | 0.8 (2) |
| C13—C8—C9—C10 | 26.79 (14) | C16—C17—C18—C21 | −179.81 (16) |
| C12—N1—C10—C11 | −1.27 (17) | C17—C18—C19—C20 | 0.8 (2) |
| C13—N1—C10—C11 | −131.91 (13) | C21—C18—C19—C20 | −178.52 (14) |
| C12—N1—C10—C9 | 120.75 (13) | C18—C19—C20—C15 | −2.2 (2) |
| C13—N1—C10—C9 | −9.89 (14) | C18—C19—C20—C13 | −178.66 (14) |

| | | | |
|---|---|---|---|
| C1—C9—C10—N1 | 115.18 (13) | C16—C15—C20—C19 | 1.9 (2) |
| C8—C9—C10—N1 | −11.22 (15) | N2—C15—C20—C19 | −176.49 (13) |
| C1—C9—C10—C11 | −125.99 (15) | C16—C15—C20—C13 | 179.15 (14) |
| C8—C9—C10—C11 | 107.60 (14) | N2—C15—C20—C13 | 0.73 (16) |
| N1—C10—C11—S1 | 32.33 (15) | N1—C13—C20—C19 | −74.82 (19) |
| C9—C10—C11—S1 | −85.63 (14) | C8—C13—C20—C19 | 50.1 (2) |
| C12—S1—C11—C10 | −41.61 (12) | C14—C13—C20—C19 | 172.50 (15) |
| C13—N1—C12—S1 | 94.75 (13) | N1—C13—C20—C15 | 108.41 (13) |
| C10—N1—C12—S1 | −30.09 (15) | C8—C13—C20—C15 | −126.64 (13) |
| C11—S1—C12—N1 | 42.32 (12) | C14—C13—C20—C15 | −4.27 (14) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C10—H10···O2 | 0.98 | 2.44 | 2.882 (2) | 107 |
| N2—H2···O3$^{i}$ | 0.86 | 2.06 | 2.903 (2) | 168 |
| C3—H3···O1$^{ii}$ | 0.93 | 2.55 | 3.302 (2) | 139 |
| C9—H9···O2$^{iii}$ | 0.98 | 2.59 | 3.320 (2) | 131 |
| C21—H21*C*···O2$^{iv}$ | 0.96 | 2.57 | 3.390 (2) | 144 |

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+1; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*, −*y*+1, −*z*+1; (iv) *x*, *y*, *z*−1.