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# *N*-[4-Acetyl-5-methyl-5-(2-*p*-tolylpropyl)-4,5-dihydro-1,3,4-thiadiazol-2-yl]acetamide

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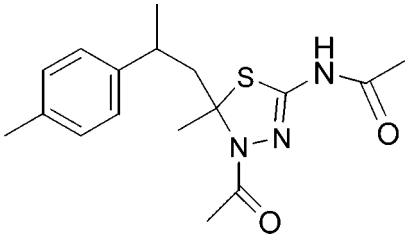
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.001 Å; $R$ factor = 0.035; $wR$ factor = 0.108; data-to-parameter ratio = 37.5.

The title heterocyclic compound, $C_{17}H_{23}N_3O_2S$, was synthesized from 4-(4-methylcyclohex-3-enyl)pent-3-en-2-one, which was isolated from *Cedrus atlantica* essential oil. The thiadiazole ring adopts a flattened envelope conformation, with the flap $sp^3$-hybridized C atom lying 0.259 (1) Å out of the plane of the other four atoms. The screw-related molecules are linked into chains along the $b$ axis by intermolecular N—H···O hydrogen bonds.

## Related literature

For 1,3,4-thiadiazole derivatives and their biological activity, see: Beatriz *et al.* (2002); Loughzail *et al.* (2009); Mazoir *et al.* (2008); Mohammed *et al.* (2008); Nakagawa *et al.* (1996); Sakthivel *et al.* (2008); Tehranchian *et al.* (2005); Wang *et al.* (1999, 2004). For puckering parameters, see: Cremer & Pople (1975).



## Experimental

### Crystal data

$C_{17}H_{23}N_3O_2S$
$M_r$ = 333.44
Monoclinic, $P2_1/n$
$a$ = 9.3984 (2) Å
$b$ = 11.0510 (2) Å
$c$ = 16.6045 (3) Å
$\beta$ = 90.442 (10)°
$V$ = 1724.52 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.20 mm$^{-1}$
$T$ = 298 (2) K
0.5 × 0.4 × 0.3 mm

### Data collection

Bruker X8 APEX CCD area-detector diffractometer
Absorption correction: none
52162 measured reflections
8286 independent reflections
7182 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.019

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.108
$S$ = 1.03
8286 reflections
221 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.51 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H4···O2$^{i}$ | 0.89 (1) | 1.96 (1) | 2.8391 (7) | 169 (1) |

Symmetry code: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2005); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia,1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2746).

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# supporting information

*Acta Cryst.* (2009). E**65**, o267–o268 [doi:10.1107/S1600536809000191]

## *N*-[4-Acetyl-5-methyl-5-(2-*p*-tolylpropyl)-4,5-dihydro-1,3,4-thiadiazol-2-yl]acetamide

**Mohamed Tebaa, Noureddine Mazoir, Celia M. Maya, Bouhmaida Nouzha, Ahmed Benharref and Moha Berraho**

### S1. Comment

1,3,4-Thiadiazole derivatives (Sakthivel *et al.*, 2008) represent an interesting class of compounds possessing diverses activities: biological (Nakagawa *et al.*, 1996), fungicidal (Wang *et al.*, 1999, 2004) and bactericidal properties (Tehranchian *et al.*, 2005). The work of our research group focused on the phytochemical study of Moroccan plants and aimed to find out new compounds, which could be used as precursors or intermediates for the synthesis of high added value specimens (Mazoir *et al.*, 2008; Loughzail *et al.*,2009). In this way, we have investigated native Cedrus species rich on sesquiterpene derivatives. Thus a new compound was obtained through chemical modification of 4-(4-methylcyclohex-3-enyl)pent-3-en-2-one, which was isolated from Cedrus Atlantica essential oil. The aromatization of the above compound followed by condensation with thiosemicarbazide (Beatriz *et al.*, 2002; Mohammed *et al.*, 2008) ending with treatment of acetic anhydride in the presence of pyridine yielded a diasterioisomers in high stereoselectivity.

The molecular structure of the title compound is shown in Fig. 1. The thiadiazole ring adopts a flattened envelop conformation as indicated by Cremer & Pople (1975) puckering parameters Q = 0.1578 (6) Å and $\varphi$ = 148.3 (2)°. Atom C5 deviates from the mean plane through other four atoms in the ring by 0.259 (1) Å.

In the crystal structure, molecules are linked into chains (Fig. 2) running along the *b* axis by intermolecular N—H···O hydrogen bonds (Table 1) involving the carbonyl and the acetamide groups.

### S2. Experimental

A solution of 4-(4-methylcyclohex-3-enyl)pent-3-en-2-one (0.5 g, 2.8 mmol) and Pd/C (10%) was heated at 423 K for 12 h. The product obtained was treated with equimolecular quantity of thiosemicarbazide and several drops of HCl (cc) were added. The reaction mixture was heated at reflux in ethanol for 6 h and then evaporated under reduced pressure and the residue obtained was purified on silica gel column using hexane-ethyl acetate (96:4) as an eluent. 0.25 mmol of the thiosemicarbazone obtained was dissolved in 3 ml of pyridine and 3 ml of acetic anhydride. The mixture was heated on a water bath for 1.5 h. The resulting residue was concentrated *in vacuo* and chromatographied on silica gel column with hexane-ethyl acetate (92:8) as an eluent. Suitable crystals were obtained by evaporation of ethyl acetate solution at 277 K.

### S3. Refinement

Atoms H4 and H7 were located in a difference map and refined freely (C7—H7 = 0.974 (11) Å and N1—H4 = 0.889 (13) Å). The remaining H atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic), 0.96 Å (methyl), 0.97 Å (methylene), 0.98Å (methine) with $U_{iso}$(H) = 1.2$U_{eq}$(aromatic, methylene, methine) or $U_{iso}$(H) = 1.5$U_{eq}$(methyl). The highest residual density peak is located 0.62 Å from atom C2 and the deepest hole is located 0.39 Å from atom H70′.

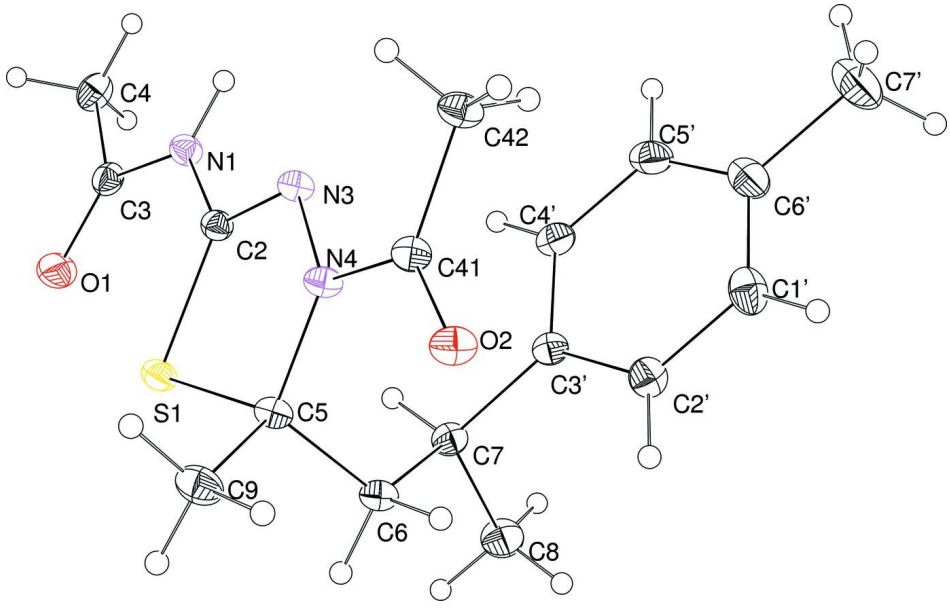


**Figure 1**

Molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

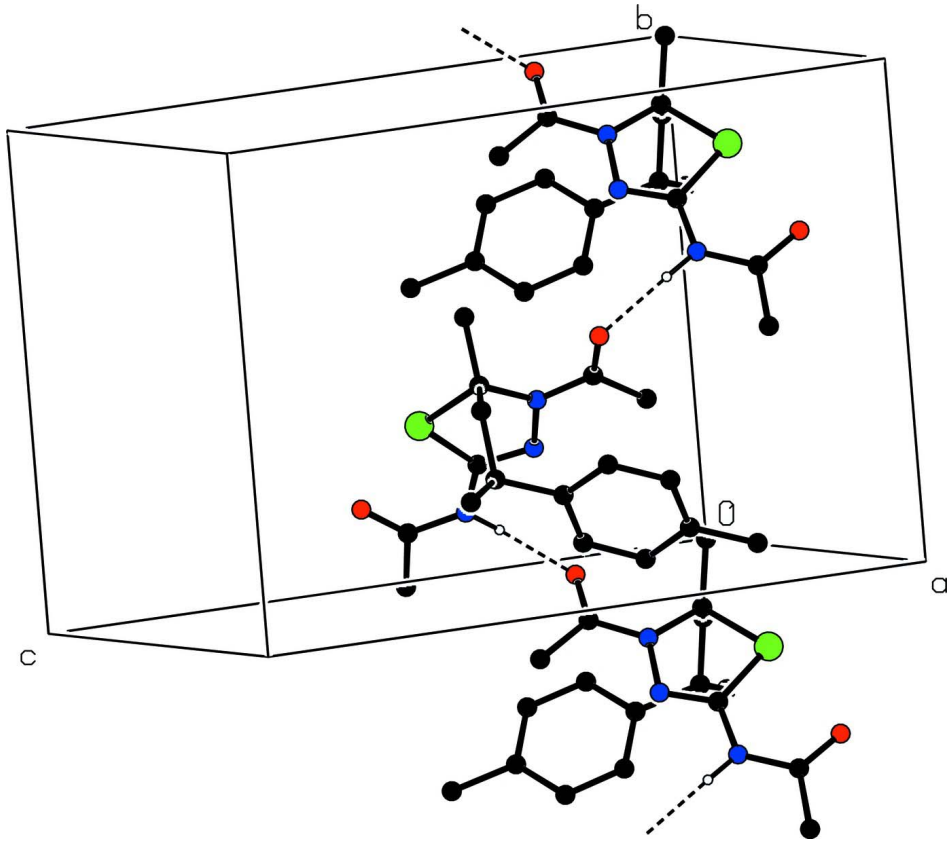


**Figure 2**

Partial packing view showing N—H···O hydrogen-bonded (dashed lines) chain running along the *b* axis. H atoms not involved in hydrogen bonding have been omitted for clarity.

### *N*-[4-Acetyl-5-methyl-5-(2-*p*-tolylpropyl)-4,5-dihydro-1,3,4- thiadiazol-2-yl]acetamide

*Crystal data*

$C_{17}H_{23}N_3O_2S$
$M_r = 333.44$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 9.3984\ (2)$ Å
$b = 11.0510\ (2)$ Å
$c = 16.6045\ (3)$ Å
$\beta = 90.442\ (10)°$
$V = 1724.52\ (6)$ Å$^3$
$Z = 4$

$F(000) = 712$
$D_x = 1.284$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 31976 reflections
$\theta = 2.2–36.5°$
$\mu = 0.20$ mm$^{-1}$
$T = 298$ K
Prism, colourless
0.5 × 0.4 × 0.3 mm

*Data collection*

Bruker X8 APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
52162 measured reflections
8286 independent reflections

7182 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 36.8°$, $\theta_{min} = 2.2°$
$h = -14\rightarrow15$
$k = -18\rightarrow17$
$l = -27\rightarrow27$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.108$
$S = 1.03$
8286 reflections
221 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0594P)^2 + 0.2773P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.51$ e Å$^{-3}$
$\Delta\rho_{min} = -0.19$ e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1′ | −0.18648 (9) | 0.69929 (8) | 0.22865 (5) | 0.03405 (16) |
| H1′ | −0.2631 | 0.7423 | 0.2493 | 0.041* |
| C2′ | −0.12028 (8) | 0.74007 (7) | 0.15872 (5) | 0.02883 (13) |
| H2′ | −0.1553 | 0.8087 | 0.1329 | 0.035* |
| C2 | 0.40705 (7) | 0.72005 (6) | 0.13520 (4) | 0.02074 (10) |
| C3′ | −0.00246 (7) | 0.68003 (6) | 0.12659 (4) | 0.02299 (11) |
| C3 | 0.58917 (7) | 0.58558 (7) | 0.08145 (4) | 0.02531 (12) |
| C4 | 0.67040 (8) | 0.47195 (8) | 0.09881 (5) | 0.03082 (14) |
| H40 | 0.6397 | 0.4094 | 0.0625 | 0.046* |
| H41 | 0.6534 | 0.4471 | 0.1533 | 0.046* |
| H42 | 0.7702 | 0.4865 | 0.0917 | 0.046* |
| C4′ | 0.04426 (8) | 0.57608 (7) | 0.16569 (5) | 0.02818 (13) |
| H4′ | 0.1221 | 0.5340 | 0.1458 | 0.034* |
| C5 | 0.24444 (7) | 0.89236 (6) | 0.09425 (4) | 0.02261 (11) |
| C5′ | −0.02462 (10) | 0.53384 (8) | 0.23496 (5) | 0.03469 (17) |
| H5′ | 0.0073 | 0.4631 | 0.2595 | 0.042* |
| C6 | 0.10835 (8) | 0.85717 (6) | 0.04946 (4) | 0.02444 (12) |
| H61 | 0.1172 | 0.8834 | −0.0060 | 0.029* |
| H62 | 0.0302 | 0.9021 | 0.0728 | 0.029* |
| C6′ | −0.13955 (10) | 0.59549 (9) | 0.26777 (5) | 0.03553 (17) |
| C7′ | −0.21196 (14) | 0.55330 (13) | 0.34392 (6) | 0.0557 (3) |
| H70′ | −0.1661 | 0.4813 | 0.3634 | 0.083* |
| H71′ | −0.3103 | 0.5363 | 0.3325 | 0.083* |
| H72′ | −0.2054 | 0.6155 | 0.3841 | 0.083* |
| C7 | 0.06784 (7) | 0.72237 (6) | 0.04914 (4) | 0.02407 (11) |
| C8 | −0.02971 (10) | 0.69513 (9) | −0.02232 (5) | 0.03593 (17) |
| H80 | 0.0164 | 0.7187 | −0.0713 | 0.054* |
| H81 | −0.1169 | 0.7395 | −0.0168 | 0.054* |
| H82 | −0.0501 | 0.6100 | −0.0239 | 0.054* |
| C9 | 0.27064 (10) | 1.02844 (7) | 0.08864 (5) | 0.03188 (15) |
| H90 | 0.3533 | 1.0493 | 0.1200 | 0.048* |
| H91 | 0.1895 | 1.0711 | 0.1090 | 0.048* |
| H92 | 0.2855 | 1.0505 | 0.0334 | 0.048* |

| | | | | |
|---|---|---|---|---|
| C41 | 0.15568 (7) | 0.88983 (6) | 0.23768 (4) | 0.02220 (11) |
| C42 | 0.16402 (9) | 0.82781 (7) | 0.31792 (4) | 0.02797 (13) |
| H420 | 0.1094 | 0.7544 | 0.3161 | 0.042* |
| H421 | 0.1265 | 0.8802 | 0.3587 | 0.042* |
| H422 | 0.2615 | 0.8091 | 0.3304 | 0.042* |
| N1 | 0.49426 (6) | 0.61997 (5) | 0.14026 (3) | 0.02274 (10) |
| N3 | 0.32596 (6) | 0.74595 (5) | 0.19539 (3) | 0.02162 (10) |
| N4 | 0.24397 (6) | 0.84788 (5) | 0.17882 (3) | 0.02167 (10) |
| O1 | 0.60479 (7) | 0.64374 (7) | 0.01970 (4) | 0.03731 (14) |
| O2 | 0.07229 (6) | 0.97409 (5) | 0.22442 (3) | 0.02734 (10) |
| S1 | 0.400452 (18) | 0.813333 (16) | 0.050251 (10) | 0.02473 (5) |
| H4 | 0.4838 (13) | 0.5774 (12) | 0.1853 (8) | 0.036 (3)* |
| H7 | 0.1540 (12) | 0.6750 (10) | 0.0411 (7) | 0.028 (3)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1′ | 0.0346 (4) | 0.0368 (4) | 0.0309 (3) | −0.0069 (3) | 0.0063 (3) | −0.0080 (3) |
| C2′ | 0.0302 (3) | 0.0267 (3) | 0.0297 (3) | 0.0019 (2) | 0.0030 (2) | −0.0024 (2) |
| C2 | 0.0225 (2) | 0.0212 (2) | 0.0185 (2) | −0.00085 (19) | −0.00014 (18) | 0.00030 (19) |
| C3′ | 0.0255 (3) | 0.0214 (3) | 0.0220 (3) | 0.0002 (2) | −0.0027 (2) | −0.00210 (19) |
| C3 | 0.0203 (2) | 0.0334 (3) | 0.0222 (3) | 0.0008 (2) | 0.00024 (19) | −0.0032 (2) |
| C4 | 0.0255 (3) | 0.0331 (3) | 0.0338 (3) | 0.0052 (2) | −0.0005 (2) | −0.0081 (3) |
| C4′ | 0.0296 (3) | 0.0246 (3) | 0.0303 (3) | −0.0003 (2) | −0.0072 (2) | 0.0011 (2) |
| C5 | 0.0293 (3) | 0.0197 (2) | 0.0188 (2) | 0.0009 (2) | −0.0005 (2) | 0.00177 (19) |
| C5′ | 0.0396 (4) | 0.0325 (4) | 0.0318 (3) | −0.0098 (3) | −0.0116 (3) | 0.0077 (3) |
| C6 | 0.0289 (3) | 0.0245 (3) | 0.0199 (2) | 0.0025 (2) | −0.0023 (2) | 0.0024 (2) |
| C6′ | 0.0407 (4) | 0.0420 (4) | 0.0238 (3) | −0.0185 (3) | −0.0029 (3) | −0.0004 (3) |
| C7′ | 0.0633 (7) | 0.0724 (8) | 0.0313 (4) | −0.0341 (6) | 0.0034 (4) | 0.0054 (5) |
| C7 | 0.0264 (3) | 0.0251 (3) | 0.0207 (2) | 0.0018 (2) | −0.0016 (2) | −0.0032 (2) |
| C8 | 0.0402 (4) | 0.0437 (4) | 0.0238 (3) | −0.0051 (3) | −0.0066 (3) | −0.0053 (3) |
| C9 | 0.0445 (4) | 0.0202 (3) | 0.0310 (3) | −0.0016 (3) | −0.0006 (3) | 0.0036 (2) |
| C41 | 0.0276 (3) | 0.0205 (2) | 0.0185 (2) | 0.0008 (2) | −0.00094 (19) | −0.00301 (19) |
| C42 | 0.0367 (3) | 0.0281 (3) | 0.0191 (3) | 0.0049 (3) | 0.0014 (2) | 0.0006 (2) |
| N1 | 0.0240 (2) | 0.0233 (2) | 0.0209 (2) | 0.00241 (18) | 0.00268 (17) | 0.00121 (18) |
| N3 | 0.0262 (2) | 0.0202 (2) | 0.0184 (2) | 0.00288 (18) | 0.00029 (17) | 0.00064 (17) |
| N4 | 0.0281 (2) | 0.0199 (2) | 0.0171 (2) | 0.00304 (18) | −0.00031 (17) | 0.00020 (16) |
| O1 | 0.0321 (3) | 0.0550 (4) | 0.0249 (2) | 0.0069 (3) | 0.0068 (2) | 0.0066 (2) |
| O2 | 0.0344 (3) | 0.0233 (2) | 0.0244 (2) | 0.00714 (18) | −0.00082 (18) | −0.00255 (17) |
| S1 | 0.02737 (8) | 0.02713 (9) | 0.01972 (8) | 0.00035 (5) | 0.00253 (5) | 0.00433 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1′—C6′ | 1.3886 (14) | C6—C7 | 1.5375 (10) |
| C1′—C2′ | 1.3963 (12) | C6—H61 | 0.97 |
| C1′—H1′ | 0.93 | C6—H62 | 0.97 |
| C2′—C3′ | 1.4001 (10) | C6′—C7′ | 1.5142 (13) |
| C2′—H2′ | 0.93 | C7′—H70′ | 0.96 |

| | | | |
|---|---|---|---|
| C2—N3 | 1.2936 (8) | C7′—H71′ | 0.96 |
| C2—N1 | 1.3788 (9) | C7′—H72′ | 0.96 |
| C2—S1 | 1.7478 (6) | C7—C8 | 1.5240 (10) |
| C3′—C4′ | 1.3891 (10) | C7—H7 | 0.974 (11) |
| C3′—C7 | 1.5239 (10) | C8—H80 | 0.96 |
| C3—O1 | 1.2199 (9) | C8—H81 | 0.96 |
| C3—N1 | 1.3810 (9) | C8—H82 | 0.96 |
| C3—C4 | 1.4966 (11) | C9—H90 | 0.96 |
| C4—H40 | 0.96 | C9—H91 | 0.96 |
| C4—H41 | 0.96 | C9—H92 | 0.96 |
| C4—H42 | 0.96 | C41—O2 | 1.2357 (8) |
| C4′—C5′ | 1.4040 (12) | C41—N4 | 1.3679 (8) |
| C4′—H4′ | 0.93 | C41—C42 | 1.4998 (10) |
| C5—N4 | 1.4878 (8) | C42—H420 | 0.96 |
| C5—C6 | 1.5250 (10) | C42—H421 | 0.96 |
| C5—C9 | 1.5267 (10) | C42—H422 | 0.96 |
| C5—S1 | 1.8609 (7) | N1—H4 | 0.889 (13) |
| C5′—C6′ | 1.3916 (14) | N3—N4 | 1.3911 (8) |
| C5′—H5′ | 0.93 | | |

| | | | |
|---|---|---|---|
| C6′—C1′—C2′ | 120.90 (8) | C6′—C7′—H70′ | 109.5 |
| C6′—C1′—H1′ | 119.5 | C6′—C7′—H71′ | 109.5 |
| C2′—C1′—H1′ | 119.5 | H70′—C7′—H71′ | 109.5 |
| C1′—C2′—C3′ | 121.52 (7) | C6′—C7′—H72′ | 109.5 |
| C1′—C2′—H2′ | 119.2 | H70′—C7′—H72′ | 109.5 |
| C3′—C2′—H2′ | 119.2 | H71′—C7′—H72′ | 109.5 |
| N3—C2—N1 | 118.94 (6) | C3′—C7—C8 | 109.53 (6) |
| N3—C2—S1 | 118.40 (5) | C3′—C7—C6 | 113.80 (5) |
| N1—C2—S1 | 122.66 (5) | C8—C7—C6 | 109.97 (6) |
| C4′—C3′—C2′ | 117.51 (7) | C3′—C7—H7 | 108.5 (7) |
| C4′—C3′—C7 | 120.72 (6) | C8—C7—H7 | 106.4 (7) |
| C2′—C3′—C7 | 121.70 (6) | C6—C7—H7 | 108.3 (7) |
| O1—C3—N1 | 122.14 (7) | C7—C8—H80 | 109.5 |
| O1—C3—C4 | 122.65 (7) | C7—C8—H81 | 109.5 |
| N1—C3—C4 | 115.21 (6) | H80—C8—H81 | 109.5 |
| C3—C4—H40 | 109.5 | C7—C8—H82 | 109.5 |
| C3—C4—H41 | 109.5 | H80—C8—H82 | 109.5 |
| H40—C4—H41 | 109.5 | H81—C8—H82 | 109.5 |
| C3—C4—H42 | 109.5 | C5—C9—H90 | 109.5 |
| H40—C4—H42 | 109.5 | C5—C9—H91 | 109.5 |
| H41—C4—H42 | 109.5 | H90—C9—H91 | 109.5 |
| C3′—C4′—C5′ | 120.78 (8) | C5—C9—H92 | 109.5 |
| C3′—C4′—H4′ | 119.6 | H90—C9—H92 | 109.5 |
| C5′—C4′—H4′ | 119.6 | H91—C9—H92 | 109.5 |
| N4—C5—C6 | 111.56 (5) | O2—C41—N4 | 121.03 (6) |
| N4—C5—C9 | 112.62 (6) | O2—C41—C42 | 122.10 (6) |
| C6—C5—C9 | 110.89 (6) | N4—C41—C42 | 116.87 (6) |
| N4—C5—S1 | 102.92 (4) | C41—C42—H420 | 109.5 |

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|---|---|---|---|
| C6—C5—S1 | 110.44 (5) | C41—C42—H421 | 109.5 |
| C9—C5—S1 | 108.08 (5) | H420—C42—H421 | 109.5 |
| C6′—C5′—C4′ | 121.47 (8) | C41—C42—H422 | 109.5 |
| C6′—C5′—H5′ | 119.3 | H420—C42—H422 | 109.5 |
| C4′—C5′—H5′ | 119.3 | H421—C42—H422 | 109.5 |
| C5—C6—C7 | 117.10 (5) | C2—N1—C3 | 124.37 (6) |
| C5—C6—H61 | 108.0 | C2—N1—H4 | 113.9 (8) |
| C7—C6—H61 | 108.0 | C3—N1—H4 | 121.7 (8) |
| C5—C6—H62 | 108.0 | C2—N3—N4 | 110.79 (5) |
| C7—C6—H62 | 108.0 | C41—N4—N3 | 118.12 (5) |
| H61—C6—H62 | 107.3 | C41—N4—C5 | 124.65 (5) |
| C1′—C6′—C5′ | 117.78 (7) | N3—N4—C5 | 116.65 (5) |
| C1′—C6′—C7′ | 120.12 (10) | C2—S1—C5 | 89.03 (3) |
| C5′—C6′—C7′ | 122.10 (10) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H4···O2$^{i}$ | 0.89 (1) | 1.96 (1) | 2.8391 (7) | 169 (1) |

Symmetry code: (i) −*x*+1/2, *y*−1/2, −*z*+1/2.