Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure and Hirshfeld surface analysis of (*Z*)-*N*′-(4-methoxybenzo[*d*]thiazol-2-yl)-3,6-di-*p*-tolyl-1,2,4-triazine-5-carboximidamide

Yaroslav K. Shtaitz,[a,b] Dmitry S. Kopchuk,[a,b,c] Vasiliy S. Gaviko,[a,d] Nizami A. Ekberov,[e] Mehmet Akkurt,[f] Gizachew Mulugeta Manahelohe[g]* and Khudayar I. Hasanov[h]

[a]The Ural Federal University, named after the first president of Russia, B. N. Yeltsin, Mira. 19 st., 620002, Yekaterinburg, Russian Federation, [b]Postovsky Institute of Organic Synthesis, Ural Branch of the Russian Academy of Sciences, Sof'i Kovalevskoy 22 st., 620137, Yekaterinburg, Russian Federation, [c]Sirius University of Science and Technology, Olympic Avenue 1, 354340, Sirius Federal Territory, Russian Federation, [d]M. N. Mikheev Institute of Metal Physics of the Ural Branch of the Russian Academy of Sciences, Sof'i Kovalevskoy 18 st., 620108, Yekaterinburg, Russian Federation, [e]Azerbaijan State Pedagogical University, 68 Uzeyir Hajibeyov St., AZ 1000, Baku, Azerbaijan, [f]Department of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Türkiye, [g]Department of Chemistry, University of Gondar, PO Box 196, Gondar, Ethiopia, and [h]Azerbaijan Medical University, Scientific Research Centre (SRC), A. Kasumzade St. 14, AZ 1022, Baku, Azerbaijan. *Correspondence e-mail: Gizachew.Mulugeta@uog.edu.et

Received 9 July 2026
Accepted 28 July 2026

Edited by T. Akitsu, Tokyo University of Science, Japan

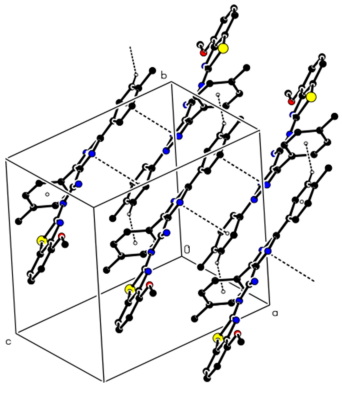
Keywords: crystal structure; 1,3-benzothiazole; 1,2,4-triazine; hydrogen bonds; Hirshfeld surface analysis.

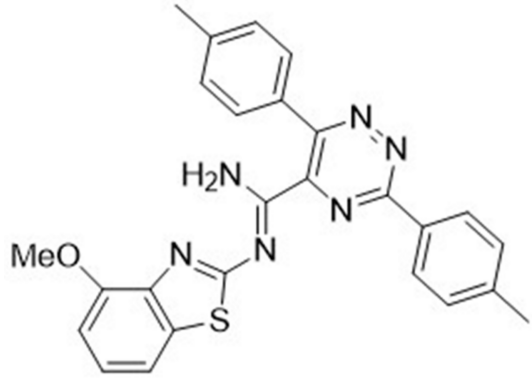
Supporting information: this article has supporting information at journals.iucr.org/e

The title molecule, $C_{26}H_{22}N_6OS$, has a stable conformation due to two intramolecular N—H···N hydrogen bonds that produce fused *S*(5) and *S*(6) rings. Layers parallel to the (010) plane are formed in the crystal by intermolecular C—H···O and C—H···N hydrogen bonding. Additionally, C—H···π and π–π [centroid-to-centroid distance = 3.7380 (12) Å] interactions link the molecules, creating ribbons along the *a*-axis direction. The intermolecular interactions were quantified using Hirshfeld surface analysis and two-dimensional fingerprint plots, which revealed the relative contributions of H···H (48.6%), C···H/H···C (18.8%), and N···H/H···N (12.0%) contacts to the crystal packing.

1. Chemical context

Heterocyclic amidine derivatives of benzothiazole are notable pharmacophores with anti-inflammatory (Sondhi *et al.*, 2011; Racane *et al.*, 2023) and neuroprotective (Anzini *et al.*, 2010) activities. In recent years, benzothiazole-triazine hybrid molecules have been reported as potent anti-tumor agents (Kumar *et al.*, 2015) and COX-2 inhibitors (Ertas *et al.*, 2022).





Previously, we have developed a method for modification of 5-cyano-1,2,4-triazines with heterocyclic amines *via* a nucleophilic *ipso*-substitution reaction (Krinochkin *et al.*, 2022; Rammohan *et al.*, 2021), thus as our next step we have tried to apply 2-aminobenzothiazoles to this methodology. It was

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

*Cg*4 is the centroid of the C13–C18 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···N4	0.89 (2)	2.17 (2)	2.602 (3)	109 (2)
N3—H3*B*···N1	0.94 (2)	1.98 (2)	2.665 (3)	128 (2)
C8—H8*C*···N5^{i}	0.96	2.60	3.532 (4)	163
C17—H17···O1^{ii}	0.93	2.60	3.492 (3)	161
C24—H24···*Cg*4^{iii}	0.93	2.96	3.796 (3)	151

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

found that solvent-free interaction of 3,6-di-*p*-tolyl-1,2,4-triazine-5-carbonitrile (**1**) with 4-methoxybenzo[*d*]thiazol-2-amine (**2**) at 423 K leads to the product (*Z*)-*N*′-(4-methoxybenzo[*d*]thiazol-2-yl)-3,6-di-p-tolyl-1,2,4-triazine-5-carboximidamide (**3**). There is six-membered intramolecular resonance-assisted hydrogen bonding in the crystal structure of (**3**), which can be used in molecular recognition (Mahmudov & Pombeiro, 2016).

2. Structural commentary

As shown Fig. 1, the conformation of the title molecule is stabilized by two intramolecular N—H···N hydrogen bonds, forming fused *S*(5) and *S*(6) rings (Table 1; Bernstein *et al.*, 1995). In a six-membered resonance-assisted hydrogen-bonding (RAHB) ring, the N3—H3*B*···N1 bond with [N···N distance = 2.663 (3) Å] falls in the N···N distance range [2.542–2.897 Å] observed for other supramolecular synthons (Gurbanov *et al.*, 2020), and indicates a low delocalization within the heterodienic moiety. The N—H···N angle of 128 (2)° differs significantly from the average O—H···O angle (149°; Bertolasi *et al.*, 1993) of *β*-diketone enols involved in similar intramolecular RAHB.

The 1,3-benzothiazole ring system (S1/N1/C1–C7) is essentially planar with an r.m.s. deviation of 0.002 Å. The least-squares plane of this ring system forms dihedral angles of 8.18 (8), 64.96 (10), and 5.33 (9)°, respectively, with the planes of the 1,2,4-triazine ring (N4–N6/C10–C12) and the benzene rings (C13–C18 and C20–C25) of its two attached 4-methylphenyl groups. The benzene rings (C13–C18 and C20–C25) of the two 4-methylphenyl groups form angles of 68.68 (12)° with each other, while they make the angles of 57.10 (11) and 11.66 (10)°, respectively, with the 1,2,4-triazine ring plane (N4–N6/C10–C12). The bond lengths and angles in the title compound are in good agreement with those reported for related compounds (see *Database survey* section).

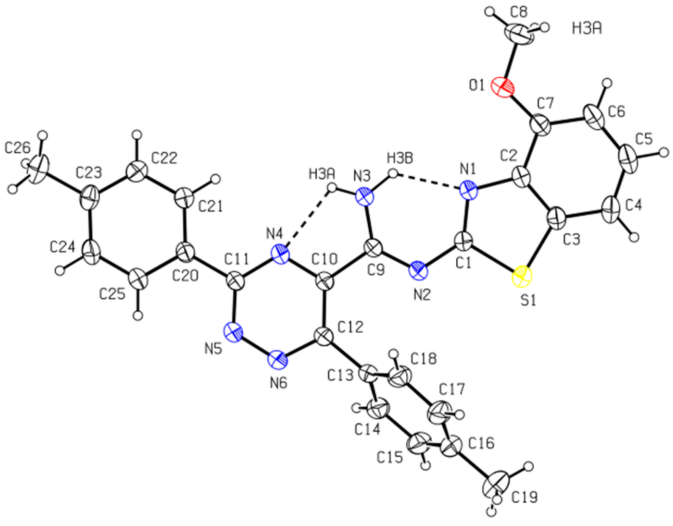


Figure 1
Molecular structure of the title compound showing the atomic labelling. Displacement ellipsoids are drawn at the 30% probability level.

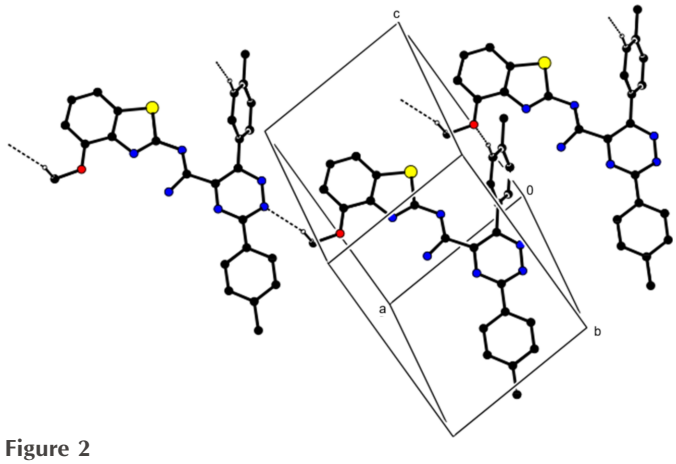


Figure 2
A partial view of the C—H···O and C—H···N bonds (dashed lines) in the unit cell. H atoms not involved in hydrogen bonding have been omitted.

3. Supramolecular features and Hirshfeld surface analysis

In the crystal, the molecules are linked by C—H···O and C—H···N hydrogen bonds, forming sheets parallel to the

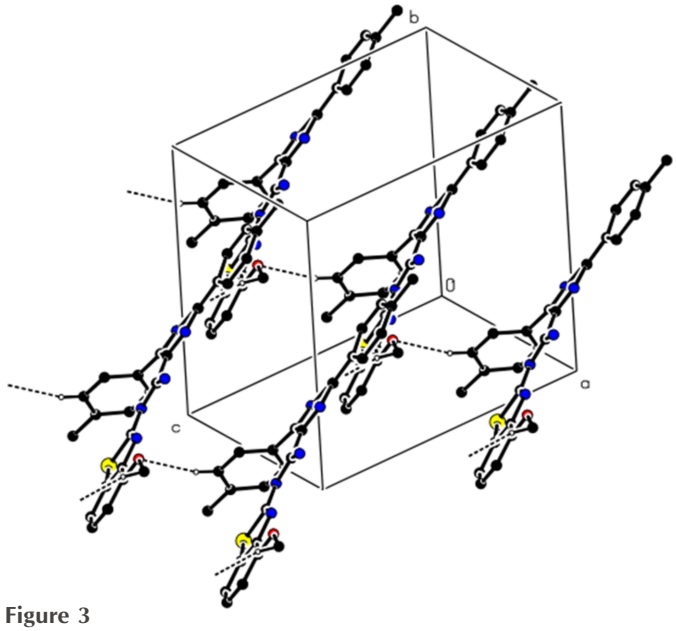


Figure 3
Crystal packing of the title compound viewed along the *a* axis showing the C—H···O and C—H···N bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

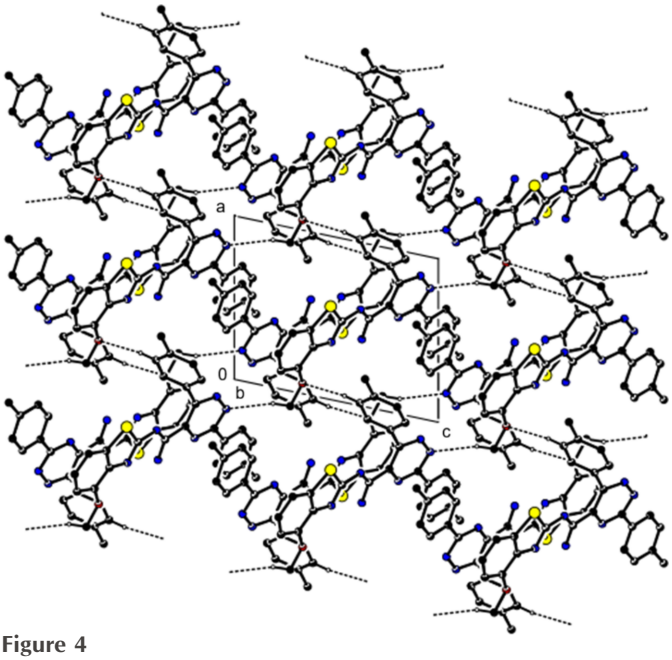


Figure 4
Crystal packing of the title compound viewed along the *b* axis showing the C—H···O and C—H···N bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

(010) plane (Figs. 2, 3 and 4; Table 1). The molecules are further connected by C—H···π and π–π interactions [$Cg2\cdots Cg5^{a}$ = 3.7380 (12) Å, slippage = 1.201 Å; symmetry code (*a*) $1-x, 1-y, -z$; where *Cg*2 and *Cg*5 are the centroids of the 1,2,4-triazine ring (N4–N6/C10–C12) and the benzene

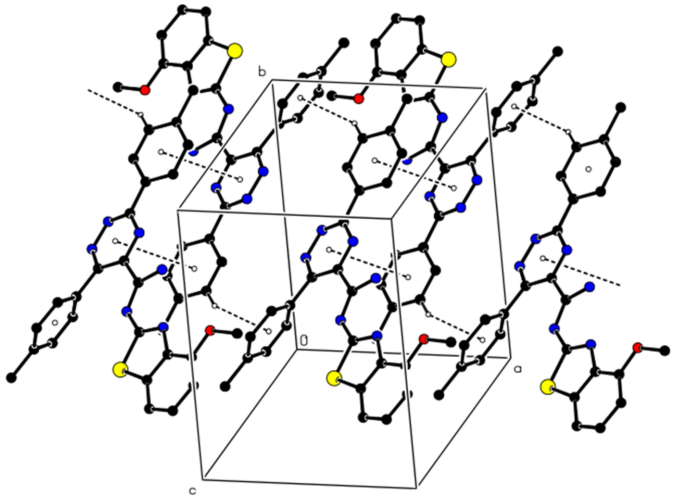


Figure 6
A view along the *c*-axis showing the C—H···π and π–π interactions (dashed lines).

ring (C20–C25) of the 4-methylphenyl group, respectively], forming ribbons along the *a*-axis direction (Figs. 5 and 6; Table 1).

A Hirshfeld surface analysis was conducted in order to confirm the existence of hydrogen bonds and intermolecular interactions (Table 2) in the crystal structure and evaluate the contributions from the various intermolecular interactions. The three-dimensional Hirshfeld surface with a d_{norm} (normalized contact distance) plot and two-dimensional

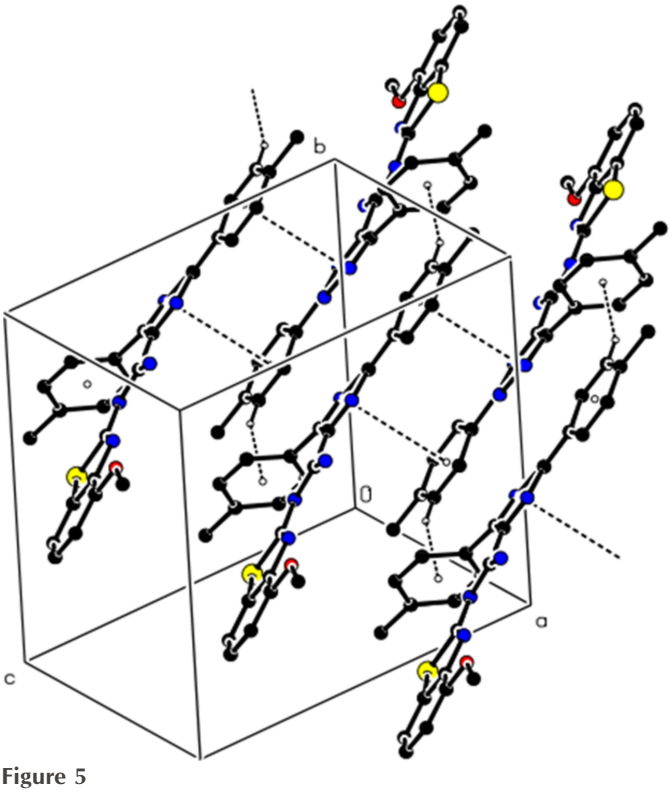


Figure 5
A view along the *a*-axis showing the C—H···π and π–π interactions (dashed lines).

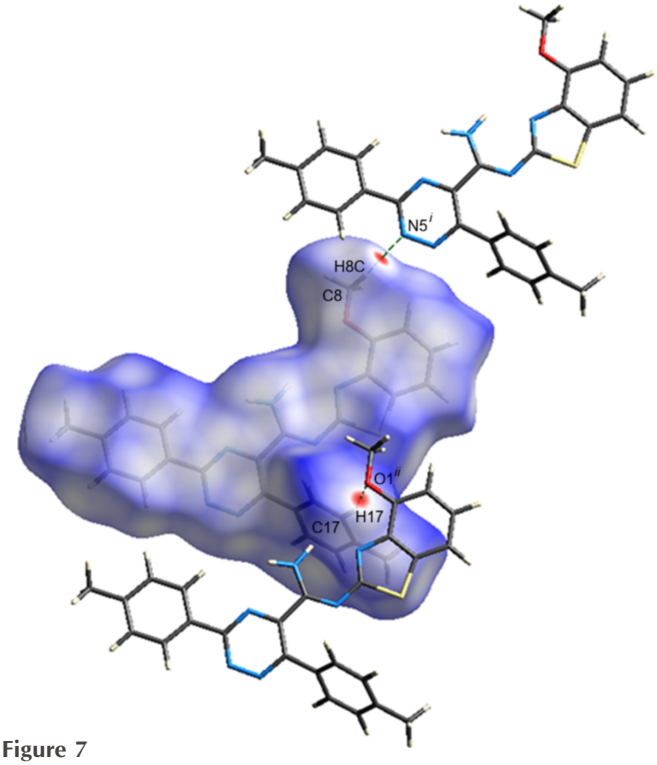


Figure 7
The Hirshfeld d_{norm} surface for the asymmetric unit with several neighboring molecules. The C—H···O and C—H···N hydrogen bonds are depicted by red dashed lines.

Table 2
Summary of short interatomic contacts (Å).

Contact	Distance	Symmetry operation
C4···S1	3.67	$1-x,-y,1-z$
H19*C*···H19*C*	2.56	$-x,-y,1-z$
O1···H17	2.60	$1+x,y,z$
H18···O1	2.87	$1-x,1-y,1-z$
H26*C*···N2	2.68	$1-x,1-y,-z$
H8*A*···H3*A*	2.53	$2-x,1-y,1-z$
H25···H8*C*	2.43	$-1+x,y,-1+z$
H25···N6	2.81	$-x,1-y,-z$
H26*B*···H4	2.51	$x,1+y,-1+z$
C26···C26	2.55	$1-x,2-y,-z$

fingerprint plots were generated with *Crystal Explorer 17.5* (Spackman *et al.*, 2021). The Hirshfeld surface was plotted over the range −0.1153 (red) to +1.3440 (blue) a.u. (Fig. 7). The red spots on the surface indicate the sites of the C8—H8*C*···N5^{i} and C17—H17···O1^{ii} interactions. The two-dimensional fingerprint plots (Fig. 8) show that the most significant contacts are H···H (48.6%), C···H/H···C (18.8%) and N···H/H···N (12.0%) interactions. Smaller contributions are made by S···H/H···S (5.4%), C···C (5.3%), N···C/C···N (4.0%), S···C/C···S (1.1%), O···C/C···O (0.8%), N···N (0.7%), O···N/N···O (0.2%) and S···S (0.1%) interactions.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 6.00, update of April 2025; Groom *et al.*, 2016) for the *N*′-(1,3-benzothiazol-2-yl)-1,2,4-triazine-5-carboximidamide unit revealed that the five compounds most closely related to the title compound are *N*-[(1,3-benzothiazol-2-yl)carbamothioyl]-4-bromobenzamide (JOMGOL: Sow *et al.*, 2024), *N*′-(1,3-benzothiazol-2-yl)benzenesulfonohydrazide (COCFOS: Baddeley *et al.*, 2019), *N*-(1,3-benzothiazol-2-yl)acetamide (TIJPAF: Nayak *et al.*, 2013), 2-[(6-methoxy-1,3-benzothiazol-2-yl)carbonoimidoyl]phenol (SUFFEG; Hijji *et al.*, 2015) and *N*-(4,6-dimethylpyrimidin-2-yl)-1,3-benzothiazol-2-amine (YAJBUI: Mohamed *et al.*, 2011).

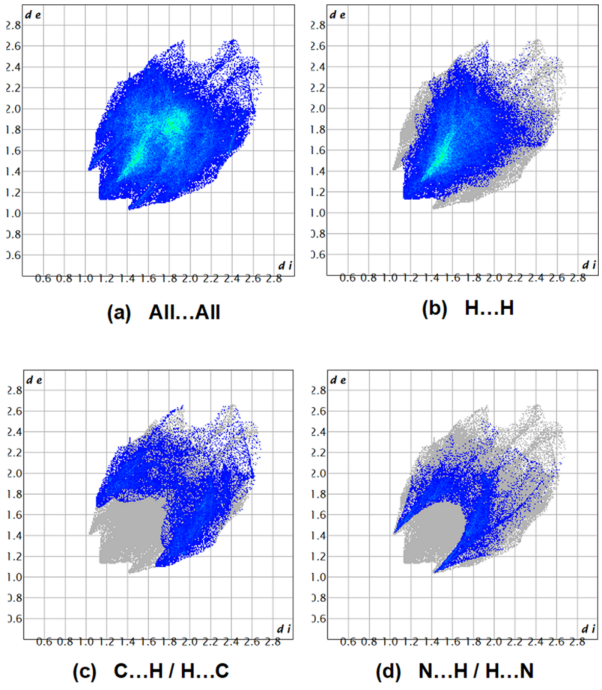


Figure 8
The two-dimensional fingerprint plots for the title compound showing (*a*) all interactions, and those delineated into (*b*) H···H, (*c*) C···H/H···C and (*d*) N···H / H···N interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

JOMGOL crystallizes in the monoclinic space group $P2_1/n$. In the crystal, pairs of adjacent molecules interact *via* intermolecular hydrogen bonds of type C—H···N, C—H···S and N—H···S, resulting in molecular layers parallel to the *ac* plane.

COCFOS crystallizes in the orthorhombic space group *Pbca*, with two molecules (*A* and *B*) in the asymmetric unit. In the crystal, molecules *A* and *B* form a supramolecular dimer by pairwise N—H···N hydrogen bonding. The dimers are assembled into chains along the *a*-axis direction by N—H···O hydrogen bonds between *A* molecules, whilst the molecules are linked along [001] by linkages between centrosymmetrically related *B* molecules. As a result, a wavy supermolecular layer is formed. C—H···O points of contact are visible as layers build along the *b* axis.

TIJPAF crystallizes in the monoclinic space group $P2_1/c$, with two molecules (*A* and *B*) in the asymmetric unit. In the crystal, pairs of N—H···N hydrogen bonds link the *A* and *B* molecules into dimers, generating $R_2^2(8)$ loops. The dimers stack along [100].

SUFFEG crystallizes in the orthorhombic space group $Pna2_1$, with two molecules in the asymmetric unit. The two independent molecules are linked by a pair of C—H···O hydrogen bonds, forming dimers with an $R_2^2(20)$ ring motif. These dimers are further linked into sheets in the *ab* plane by weak intermolecular C—H···N interactions.

YAJBUI crystallizes in the monoclinic space group $P2_1/n$. The secondary amino N atom forms an intermolecular N—H···N hydrogen bond to an N atom of the fused ring of an adjacent molecule, generating a centrosymmetric cyclic hydrogen-bonded dimer with an $R_2^2(8)$ motif.

5. Synthesis and crystallization

The starting compound 5-cyano-1,2,4-triazine was prepared according to a literature procedure (Kozhevnikov *et al.*, 2002).

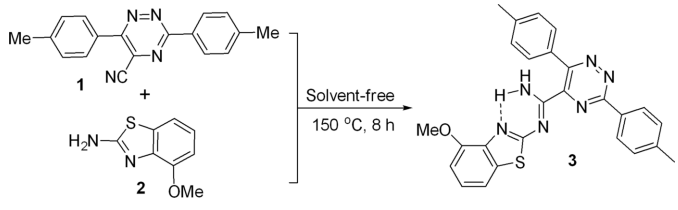


Figure 9
Synthesis of title compound.

0.37 mmol of 3,6-di-*p*-tolyl-1,2,4-triazine-5-carbonitrile (**1**) and 0.41 mmol of 4-methoxybenzo[*d*]thiazol-2-amine (**2**) were added into a round-bottom flask and the resulting mixture was heated at 423 K for 8 h under inert atmosphere. The product was isolated by column chromatography (eluent AcOEt: $CDCl_3$ = 1:9; R_f = 0.7 − 0.4) (Fig. 9). Analytical samples were obtained by recrystallization from ethanol solution. A single crystal of the title compound (**3**) was obtained by slow evaporation of its chloroform solution.

Yield 69 mg (40%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.43 (*s*, 3H, CH_3), 2.48 (*s*, 3H, CH_3), 4.02 (*s*, 3H, OCH_3), 6.85-6.91 (*m*, 1H, benzothiazole), 7.19–7.28 (*m*, 3H, $C_6H_4CH_3$+benzothiazole), 7.28–7.33 (m, 1H, benzothiazole), 7.36–7.41 (*m*, 2H, $C_6H_4CH_3$), 7.53 (*br. s*, 1H, NH), 7.69–7.75 (*m*, 2H, $C_6H_4CH_3$), 8.47–8.52 (*m*, 2H, $C_6H_4CH_3$), 10.38 (*br. s*, 1H, NH). Mass-spectrum, *m*/*z* (I_{rel}, %): *m*/*z*: 467.17 $[M+H]^+$ (100). Found, %: C 66.91, H 4.73, N 18.02. $C_{26}H_{22}N_6OS$. Calculated, %: C 66.93, H 4.75, N 18.01.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The H atoms of the NH_2 groups were found in difference-Fourier maps and were refined with U_{iso}(H) = 1.2U_{eq}(N). The C-bound H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined using a riding model, with U_{iso}(H) = 1.2 or 1.5U_{eq}(C).

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{26}H_{22}N_6OS$
M_r	466.55
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, *b*, *c* (Å)	9.0055 (1), 11.5397 (1), 11.7583 (1)
α, β, γ (°)	103.401 (1), 100.311 (1), 95.215 (1)
V (Å^3)	1158.22 (2)
Z	2
Radiation type	Mo *K*α
μ (mm^{-1})	0.17
Crystal size (mm)	0.41 × 0.28 × 0.13
Data collection	
Diffractometer	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021)
T_{min}, T_{max}	0.745, 0.978
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	176835, 6298, 3926
R_{int}	0.071
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.695
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.050, 0.180, 1.13
No. of reflections	6298
No. of parameters	316
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.33

Computer programs: *CrysAlis PRO* (Rigaku OD, 2021), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

Acknowledgements

The authors' contributions are as follows. Conceptualization, MA and GMM; synthesis, YKS, DSK and VSG; X-ray analysis, YKS, DSK and VSG; writing (review and editing of the manuscript) NAE, MA, GMM and KIH; funding acquisition, YKS, DSK, VSG, NAE and KIH; supervision, MA and GMM.

Funding information

Funding for this research was provided by: the Ministry of Science and Higher Education of the Russian Federation, the Azerbaijan State Pedagogical University and Azerbaijan Medical University (contract No. 124020200072-0).

References

Anzini, M., Chelini, A., Mancini, A., Cappelli, A., Frosini, M., Ricci, L., Valoti, M., Magistretti, J., Castelli, L., Giordani, A., Makovec, F. & Vomero, S. (2010). *J. Med. Chem.* **53**, 734–744.

Baddeley, T. C., de Souza, M. V. N., Wardell, J. L., Jotani, M. M. & Tiekink, E. R. T. (2019). *Acta Cryst.* E**75**, 516–523.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bertolasi, V., Ferretti, V., Gilli, P., Gilli, G., Issa, Y. M. & Sherif, O. E. (1993). *J. Chem. Soc. Perkin Trans. 2* pp. 2223–2228.

Ertaş, M., Biltekin, S. N., Berk, B., Yurttaş, L. & Demirayak, S. (2022). *Phosphorus Sulfur Silicon* **197**, 1123–1135.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Gurbanov, A. V., Kuznetsov, M. L., Demukhamedova, S. D., Alieva, I. N., Godjaev, N. M., Zubkov, F. I., Mahmudov, K. T. & Pombeiro, A. J. L. (2020). *CrystEngComm* **22**, 628–633.

Hijji, Y., Barare, B., Wairia, G., Butcher, R. J. & Wikaira, J. (2015). *Acta Cryst.* E**71**, 385–387.

Kozhevnikov, D. N., Kozhevnikov, V. N., Kovalev, I. S., Rusinov, V. L., Chupakhin, O. N. & Aleksandrov, G. G. (2002). *Russ. J. Org. Chem.* **38**, 744–750.

Krinochkin, A. P., Shtaitz, Y. K., Rammohan, A., Butorin, I. I., Savchuk, M. I., Khalymbadzha, I. A., Kopchuk, D. S., Slepukhin, P. A., Melekhin, V. V., Shcheglova, A. V., Zyryanov, G. V. & Chupakhin, O. N. (2022). *Eur. J. Org. Chem.* e202200227.

Kumar, G. J., Kumar, S. N., Thummuri, D., Adari, L. B. S., Naidu, V. G. M., Srinivas, K. & Rao, J. (2015). *Med. Chem. Res.* **24**, 3991–4001.

Mahmudov, K. T. & Pombeiro, A. J. L. (2016). *Chem. Eur. J.* **22**, 16356–16398.

Mohamed, S. K., Horton, P. N., El-Remaily, M. A. A., Abdel-Ghany, H. & Ng, S. W. (2011). *Acta Cryst.* E**67**, o3131.

Nayak, P. S., Narayana, B., Jasinski, J. P., Yathirajan, H. S. & Kaur, M. (2013). *Acta Cryst.* E**69**, o1622.

Racané, L., Zlatić, K., Cindrić, M., Mehić, E., Karminski–Zamola, G., Taylor, M. C., Kelly, J. M., Malić, S. R., Stojković, M. R., Kralj, M. & Hranjec, M. (2023). *ChemMedChem* **18**, e202300261.

Rammohan, A., Reddy, G. M., Krinochkin, A. P., Kopchuk, D. S., Savchuk, M. I., Shtaitz, Y. K., Zyryanov, G. V., Rusinov, V. L. & Chupakhin, O. N. (2021). *Synth. Commun.* **51**, 256–262.

Rigaku OD (2021). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Sondhi, S. M., Rani, R., Roy, P., Agrawal, S. K. & Saxena, A. K. (2011). *J. Heterocycl. Chem.* **48**, 921–926.
Sow, S., Thiam, M., Odame, F., Thiam, E. I., Diouf, O., Ellena, J., Gaye, M. & Tshentu, Z. (2024). *Acta Cryst.* E**80**, 663–666.
Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). *J. Appl. Cryst.* **54**, 1006–1011.
Spek, A. L. (2020). *Acta Cryst.* E**76**, 1–11.

supporting information

Acta Cryst. (2026). E**82** [https://doi.org/10.1107/S2056989026007760]

Crystal structure and Hirshfeld surface analysis of (*Z*)-*N*′-(4-methoxybenzo[*d*]thiazol-2-yl)-3,6-di-*p*-tolyl-1,2,4-triazine-5-carboximidamide

Yaroslav K. Shtaitz, Dmitry S. Kopchuk, Vasiliy S. Gaviko, Nizami A. Ekberov, Mehmet Akkurt, Gizachew Mulugeta Manahelohe and Khudayar I. Hasanov

Computing details

(*Z*)-*N*′-(4-Methoxybenzo[*d*]thiazol-2-yl)-3,6-di-*p*-tolyl-1,2,4-triazine-5-carboximidamide

Crystal data

$C_{26}H_{22}N_6OS$
$M_r = 466.55$
Triclinic, $P\overline{1}$
$a = 9.0055$ (1) Å
$b = 11.5397$ (1) Å
$c = 11.7583$ (1) Å
$\alpha = 103.401$ (1)°
$\beta = 100.311$ (1)°
$\gamma = 95.215$ (1)°
$V = 1158.22$ (2) Å^3
$Z = 2$
$F(000) = 488$
$D_x = 1.338$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3380 reflections
$\theta = 1.8$–25.2°
$\mu = 0.17$ mm^{-1}
$T = 293$ K
Block, yellow
0.41 × 0.28 × 0.13 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Mo) X-ray Source
Mirror monochromator
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2021)
$T_{min} = 0.745$, $T_{max} = 0.978$
176835 measured reflections
6298 independent reflections
3926 reflections with $I > 2\sigma(I)$
$R_{int} = 0.071$
$\theta_{max} = 29.6°$, $\theta_{min} = 1.8°$
$h = -12\rightarrow12$
$k = -16\rightarrow15$
$l = -15\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.180$
$S = 1.13$
6298 reflections
316 parameters
0 restraints
Primary atom site location: difference Fourier map
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 0.5317P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ 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}
C1	0.5038 (2)	0.27699 (18)	0.47268 (18)	0.0465 (4)
C2	0.6980 (2)	0.26819 (18)	0.61272 (17)	0.0466 (4)
C3	0.5968 (3)	0.1778 (2)	0.6285 (2)	0.0550 (5)
C4	0.6385 (3)	0.1141 (2)	0.7144 (2)	0.0705 (7)
H4	0.570738	0.053160	0.724153	0.085*
C5	0.7819 (3)	0.1445 (3)	0.7834 (2)	0.0748 (8)
H5	0.811968	0.102707	0.840428	0.090*
C6	0.8843 (3)	0.2356 (3)	0.7711 (2)	0.0668 (7)
H6	0.980545	0.254748	0.820683	0.080*
C7	0.8450 (2)	0.2984 (2)	0.68614 (19)	0.0530 (5)
C8	1.0894 (3)	0.4134 (3)	0.7260 (3)	0.0911 (10)
H8A	1.140292	0.479608	0.704830	0.137*
H8B	1.137588	0.343166	0.703040	0.137*
H8C	1.095260	0.432897	0.810727	0.137*
C9	0.4539 (2)	0.39486 (18)	0.33990 (16)	0.0423 (4)
C10	0.3467 (2)	0.42415 (17)	0.24019 (16)	0.0412 (4)
C11	0.3239 (2)	0.53073 (18)	0.09882 (17)	0.0431 (4)
C12	0.1965 (2)	0.36600 (19)	0.18987 (18)	0.0453 (4)
C13	0.1038 (2)	0.28046 (19)	0.23459 (18)	0.0468 (5)
C14	0.0420 (3)	0.1680 (2)	0.1617 (2)	0.0571 (5)
H14	0.059479	0.146122	0.084447	0.068*
C15	−0.0451 (3)	0.0881 (2)	0.2018 (3)	0.0662 (6)
H15	−0.082782	0.012051	0.151942	0.079*
C16	−0.0780 (3)	0.1179 (3)	0.3144 (3)	0.0667 (7)
C17	−0.0205 (3)	0.2330 (3)	0.3851 (2)	0.0717 (7)
H17	−0.044045	0.256997	0.460132	0.086*
C18	0.0705 (3)	0.3125 (2)	0.3471 (2)	0.0610 (6)
H18	0.109637	0.388064	0.397334	0.073*
C19	−0.1738 (4)	0.0310 (3)	0.3591 (3)	0.1027 (11)
H19A	−0.231054	−0.031130	0.292471	0.154*
H19B	−0.242585	0.073161	0.401488	0.154*
H19C	−0.108756	−0.004213	0.411681	0.154*
C20	0.3869 (2)	0.62587 (18)	0.04898 (18)	0.0456 (4)
C21	0.5380 (2)	0.67711 (19)	0.0873 (2)	0.0516 (5)
H21	0.600772	0.652539	0.146502	0.062*
C22	0.5965 (3)	0.7648 (2)	0.0380 (2)	0.0571 (5)
H22	0.697977	0.798796	0.065626	0.069*
C23	0.5077 (3)	0.80274 (19)	−0.0510 (2)	0.0545 (5)
C24	0.3563 (3)	0.7517 (2)	−0.0881 (2)	0.0600 (6)

H24	0.294026	0.776157	−0.147659	0.072*
C25	0.2957 (3)	0.6656 (2)	−0.0388 (2)	0.0544 (5)
H25	0.193187	0.633915	−0.064515	0.065*
C26	0.5720 (4)	0.8962 (2)	−0.1063 (3)	0.0733 (7)
H26A	0.652934	0.949887	−0.048577	0.110*
H26B	0.493120	0.941006	−0.131536	0.110*
H26C	0.610847	0.857396	−0.174114	0.110*
N1	0.64364 (19)	0.32416 (15)	0.52443 (14)	0.0455 (4)
N2	0.40818 (19)	0.30594 (16)	0.38132 (15)	0.0494 (4)
N3	0.5885 (2)	0.46419 (19)	0.37481 (17)	0.0542 (5)
H3A	0.598 (3)	0.527 (2)	0.344 (2)	0.065*
H3B	0.664 (3)	0.445 (2)	0.431 (2)	0.065*
N4	0.40762 (18)	0.50838 (15)	0.19569 (14)	0.0440 (4)
N5	0.1895 (2)	0.46715 (18)	0.04002 (16)	0.0560 (5)
N6	0.12501 (19)	0.38601 (18)	0.08723 (16)	0.0544 (5)
O1	0.93428 (18)	0.39028 (16)	0.66585 (15)	0.0650 (4)
S1	0.42638 (7)	0.16195 (6)	0.52779 (6)	0.0669 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0459 (10)	0.0492 (11)	0.0447 (10)	0.0013 (8)	0.0035 (8)	0.0194 (9)
C2	0.0510 (11)	0.0504 (11)	0.0405 (10)	0.0120 (9)	0.0056 (8)	0.0165 (8)
C3	0.0611 (13)	0.0542 (12)	0.0529 (12)	0.0088 (10)	0.0064 (10)	0.0236 (10)
C4	0.0857 (18)	0.0673 (15)	0.0701 (16)	0.0128 (13)	0.0137 (14)	0.0415 (13)
C5	0.0890 (19)	0.0844 (19)	0.0641 (15)	0.0310 (16)	0.0092 (14)	0.0424 (14)
C6	0.0663 (15)	0.0840 (18)	0.0549 (13)	0.0268 (13)	0.0011 (11)	0.0295 (12)
C7	0.0496 (11)	0.0643 (13)	0.0455 (11)	0.0128 (10)	0.0051 (9)	0.0168 (10)
C8	0.0507 (14)	0.134 (3)	0.0775 (19)	−0.0033 (16)	−0.0105 (13)	0.0300 (19)
C9	0.0414 (10)	0.0470 (10)	0.0365 (9)	0.0014 (8)	0.0021 (7)	0.0128 (8)
C10	0.0391 (9)	0.0468 (10)	0.0378 (9)	0.0047 (8)	0.0047 (7)	0.0139 (8)
C11	0.0391 (9)	0.0503 (11)	0.0415 (10)	0.0079 (8)	0.0040 (7)	0.0176 (8)
C12	0.0391 (9)	0.0542 (11)	0.0433 (10)	0.0041 (8)	0.0044 (8)	0.0175 (9)
C13	0.0351 (9)	0.0580 (12)	0.0480 (11)	0.0033 (8)	0.0030 (8)	0.0203 (9)
C14	0.0505 (12)	0.0634 (14)	0.0570 (13)	0.0031 (10)	0.0116 (10)	0.0163 (11)
C15	0.0571 (14)	0.0591 (14)	0.0802 (17)	−0.0023 (11)	0.0105 (12)	0.0201 (12)
C16	0.0510 (13)	0.0774 (17)	0.0797 (17)	0.0009 (11)	0.0124 (12)	0.0397 (14)
C17	0.0647 (15)	0.095 (2)	0.0580 (14)	−0.0052 (14)	0.0196 (12)	0.0257 (14)
C18	0.0558 (13)	0.0708 (15)	0.0534 (13)	−0.0038 (11)	0.0129 (10)	0.0133 (11)
C19	0.093 (2)	0.108 (3)	0.114 (3)	−0.019 (2)	0.025 (2)	0.053 (2)
C20	0.0468 (10)	0.0481 (11)	0.0440 (10)	0.0097 (8)	0.0061 (8)	0.0167 (8)
C21	0.0478 (11)	0.0544 (12)	0.0556 (12)	0.0059 (9)	0.0056 (9)	0.0241 (10)
C22	0.0528 (12)	0.0550 (12)	0.0649 (14)	0.0020 (10)	0.0104 (10)	0.0212 (11)
C23	0.0694 (14)	0.0447 (11)	0.0591 (13)	0.0169 (10)	0.0240 (11)	0.0210 (10)
C24	0.0693 (15)	0.0609 (13)	0.0585 (13)	0.0197 (11)	0.0097 (11)	0.0312 (11)
C25	0.0497 (11)	0.0603 (13)	0.0559 (12)	0.0097 (10)	0.0020 (9)	0.0263 (10)
C26	0.097 (2)	0.0598 (14)	0.0801 (17)	0.0171 (13)	0.0397 (16)	0.0333 (13)
N1	0.0438 (9)	0.0511 (9)	0.0421 (9)	0.0035 (7)	0.0017 (7)	0.0195 (7)

N2	0.0452 (9)	0.0546 (10)	0.0455 (9)	−0.0020 (7)	−0.0034 (7)	0.0205 (8)
N3	0.0424 (9)	0.0663 (12)	0.0549 (11)	−0.0050 (8)	−0.0041 (8)	0.0331 (9)
N4	0.0444 (9)	0.0481 (9)	0.0401 (8)	0.0061 (7)	0.0034 (7)	0.0163 (7)
N5	0.0466 (10)	0.0694 (12)	0.0536 (10)	−0.0001 (8)	−0.0016 (8)	0.0311 (9)
N6	0.0412 (9)	0.0695 (12)	0.0534 (10)	−0.0004 (8)	−0.0007 (7)	0.0283 (9)
O1	0.0477 (9)	0.0818 (12)	0.0610 (10)	−0.0005 (8)	−0.0061 (7)	0.0260 (9)
S1	0.0618 (4)	0.0670 (4)	0.0726 (4)	−0.0104 (3)	−0.0026 (3)	0.0391 (3)

Geometric parameters (Å, º)

C1—N1	1.304 (2)	C13—C18	1.383 (3)
C1—N2	1.377 (3)	C14—C15	1.376 (3)
C1—S1	1.750 (2)	C14—H14	0.9300
C2—N1	1.386 (2)	C15—C16	1.381 (4)
C2—C3	1.389 (3)	C15—H15	0.9300
C2—C7	1.410 (3)	C16—C17	1.390 (4)
C3—C4	1.400 (3)	C16—C19	1.508 (4)
C3—S1	1.729 (2)	C17—C18	1.377 (3)
C4—C5	1.363 (4)	C17—H17	0.9300
C4—H4	0.9300	C18—H18	0.9300
C5—C6	1.385 (4)	C19—H19A	0.9600
C5—H5	0.9300	C19—H19B	0.9600
C6—C7	1.379 (3)	C19—H19C	0.9600
C6—H6	0.9300	C20—C21	1.383 (3)
C7—O1	1.363 (3)	C20—C25	1.392 (3)
C8—O1	1.419 (3)	C21—C22	1.385 (3)
C8—H8A	0.9600	C21—H21	0.9300
C8—H8B	0.9600	C22—C23	1.379 (3)
C8—H8C	0.9600	C22—H22	0.9300
C9—N2	1.299 (3)	C23—C24	1.385 (3)
C9—N3	1.331 (2)	C23—C26	1.506 (3)
C9—C10	1.503 (3)	C24—C25	1.379 (3)
C10—N4	1.327 (2)	C24—H24	0.9300
C10—C12	1.412 (3)	C25—H25	0.9300
C11—N5	1.336 (3)	C26—H26A	0.9600
C11—N4	1.340 (2)	C26—H26B	0.9600
C11—C20	1.475 (3)	C26—H26C	0.9600
C12—N6	1.343 (3)	N3—H3A	0.89 (3)
C12—C13	1.482 (3)	N3—H3B	0.93 (3)
C13—C14	1.383 (3)	N5—N6	1.334 (2)
N1—C1—N2	128.93 (18)	C15—C16—C19	122.0 (3)
N1—C1—S1	115.58 (15)	C17—C16—C19	121.0 (3)
N2—C1—S1	115.49 (15)	C18—C17—C16	121.8 (2)
N1—C2—C3	116.05 (18)	C18—C17—H17	119.1
N1—C2—C7	124.46 (19)	C16—C17—H17	119.1
C3—C2—C7	119.48 (19)	C17—C18—C13	120.3 (2)
C2—C3—C4	121.5 (2)	C17—C18—H18	119.8

C2—C3—S1	109.14 (15)	C13—C18—H18	119.8
C4—C3—S1	129.4 (2)	C16—C19—H19A	109.5
C5—C4—C3	117.7 (2)	C16—C19—H19B	109.5
C5—C4—H4	121.1	H19A—C19—H19B	109.5
C3—C4—H4	121.1	C16—C19—H19C	109.5
C4—C5—C6	122.1 (2)	H19A—C19—H19C	109.5
C4—C5—H5	119.0	H19B—C19—H19C	109.5
C6—C5—H5	119.0	C21—C20—C25	118.3 (2)
C7—C6—C5	120.7 (2)	C21—C20—C11	121.12 (17)
C7—C6—H6	119.6	C25—C20—C11	120.58 (19)
C5—C6—H6	119.6	C20—C21—C22	120.5 (2)
O1—C7—C6	126.5 (2)	C20—C21—H21	119.8
O1—C7—C2	115.05 (18)	C22—C21—H21	119.8
C6—C7—C2	118.5 (2)	C23—C22—C21	121.6 (2)
O1—C8—H8A	109.5	C23—C22—H22	119.2
O1—C8—H8B	109.5	C21—C22—H22	119.2
H8A—C8—H8B	109.5	C22—C23—C24	117.5 (2)
O1—C8—H8C	109.5	C22—C23—C26	121.6 (2)
H8A—C8—H8C	109.5	C24—C23—C26	120.8 (2)
H8B—C8—H8C	109.5	C25—C24—C23	121.6 (2)
N2—C9—N3	127.22 (18)	C25—C24—H24	119.2
N2—C9—C10	118.00 (17)	C23—C24—H24	119.2
N3—C9—C10	114.77 (17)	C24—C25—C20	120.4 (2)
N4—C10—C12	120.41 (17)	C24—C25—H25	119.8
N4—C10—C9	113.95 (16)	C20—C25—H25	119.8
C12—C10—C9	125.54 (17)	C23—C26—H26A	109.5
N5—C11—N4	123.82 (18)	C23—C26—H26B	109.5
N5—C11—C20	117.49 (17)	H26A—C26—H26B	109.5
N4—C11—C20	118.57 (17)	C23—C26—H26C	109.5
N6—C12—C10	118.44 (18)	H26A—C26—H26C	109.5
N6—C12—C13	113.36 (17)	H26B—C26—H26C	109.5
C10—C12—C13	128.18 (17)	C1—N1—C2	110.04 (17)
C14—C13—C18	118.3 (2)	C9—N2—C1	119.35 (17)
C14—C13—C12	120.26 (19)	C9—N3—H3A	115.9 (17)
C18—C13—C12	121.4 (2)	C9—N3—H3B	118.7 (16)
C15—C14—C13	120.8 (2)	H3A—N3—H3B	125 (2)
C15—C14—H14	119.6	C10—N4—C11	117.28 (17)
C13—C14—H14	119.6	N6—N5—C11	118.75 (17)
C14—C15—C16	121.6 (2)	N5—N6—C12	120.45 (17)
C14—C15—H15	119.2	C7—O1—C8	117.1 (2)
C16—C15—H15	119.2	C3—S1—C1	89.20 (10)
C15—C16—C17	117.0 (2)		

N1—C2—C3—C4	179.6 (2)	N4—C11—C20—C21	11.0 (3)
C7—C2—C3—C4	−1.3 (4)	N5—C11—C20—C25	14.2 (3)
N1—C2—C3—S1	0.3 (2)	N4—C11—C20—C25	−169.59 (19)
C7—C2—C3—S1	179.36 (17)	C25—C20—C21—C22	−0.7 (3)
C2—C3—C4—C5	0.6 (4)	C11—C20—C21—C22	178.7 (2)

S1—C3—C4—C5	179.9 (2)	C20—C21—C22—C23	−0.8 (4)
C3—C4—C5—C6	0.6 (4)	C21—C22—C23—C24	1.3 (4)
C4—C5—C6—C7	−1.1 (4)	C21—C22—C23—C26	−178.8 (2)
C5—C6—C7—O1	179.3 (2)	C22—C23—C24—C25	−0.3 (4)
C5—C6—C7—C2	0.5 (4)	C26—C23—C24—C25	179.7 (2)
N1—C2—C7—O1	0.7 (3)	C23—C24—C25—C20	−1.1 (4)
C3—C2—C7—O1	−178.3 (2)	C21—C20—C25—C24	1.6 (3)
N1—C2—C7—C6	179.7 (2)	C11—C20—C25—C24	−177.8 (2)
C3—C2—C7—C6	0.7 (3)	N2—C1—N1—C2	179.9 (2)
N2—C9—C10—N4	−173.89 (18)	S1—C1—N1—C2	0.6 (2)
N3—C9—C10—N4	5.3 (3)	C3—C2—N1—C1	−0.5 (3)
N2—C9—C10—C12	2.6 (3)	C7—C2—N1—C1	−179.6 (2)
N3—C9—C10—C12	−178.2 (2)	N3—C9—N2—C1	1.0 (3)
N4—C10—C12—N6	8.8 (3)	C10—C9—N2—C1	−179.96 (18)
C9—C10—C12—N6	−167.53 (19)	N1—C1—N2—C9	−3.2 (4)
N4—C10—C12—C13	−172.7 (2)	S1—C1—N2—C9	176.18 (16)
C9—C10—C12—C13	11.0 (3)	C12—C10—N4—C11	−3.2 (3)
N6—C12—C13—C14	54.0 (3)	C9—C10—N4—C11	173.53 (17)
C10—C12—C13—C14	−124.6 (2)	N5—C11—N4—C10	−5.5 (3)
N6—C12—C13—C18	−122.7 (2)	C20—C11—N4—C10	178.55 (17)
C10—C12—C13—C18	58.7 (3)	N4—C11—N5—N6	8.4 (3)
C18—C13—C14—C15	−2.7 (3)	C20—C11—N5—N6	−175.61 (19)
C12—C13—C14—C15	−179.6 (2)	C11—N5—N6—C12	−2.2 (3)
C13—C14—C15—C16	2.0 (4)	C10—C12—N6—N5	−5.9 (3)
C14—C15—C16—C17	0.7 (4)	C13—C12—N6—N5	175.36 (19)
C14—C15—C16—C19	179.8 (3)	C6—C7—O1—C8	9.3 (4)
C15—C16—C17—C18	−2.6 (4)	C2—C7—O1—C8	−171.8 (2)
C19—C16—C17—C18	178.3 (3)	C2—C3—S1—C1	0.05 (17)
C16—C17—C18—C13	1.8 (4)	C4—C3—S1—C1	−179.3 (3)
C14—C13—C18—C17	0.9 (4)	N1—C1—S1—C3	−0.38 (18)
C12—C13—C18—C17	177.7 (2)	N2—C1—S1—C3	−179.81 (18)
N5—C11—C20—C21	−165.3 (2)		

Hydrogen-bond geometry (Å, º)

*Cg*4 is the centroid of the C13–C18 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···N4	0.89 (2)	2.17 (2)	2.602 (3)	109 (2)
N3—H3*B*···N1	0.94 (2)	1.98 (2)	2.665 (3)	128 (2)
C8—H8*C*···N5^{i}	0.96	2.60	3.532 (4)	163
C17—H17···O1^{ii}	0.93	2.60	3.492 (3)	161
C21—H21···N4	0.93	2.53	2.840 (3)	100
C24—H24···*Cg*4^{iii}	0.93	2.96	3.796 (3)	151

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