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Syntheses and crystal structures of two adamantyl-substituted 1,2,4-triazole-5-thione *N*-Mannich bases

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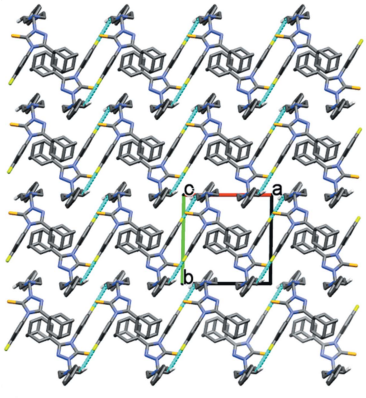
Supporting information: this article has supporting information at journals.iucr.org/e

In the title *N*-Mannich bases, 3-(adamantan-1-yl)-4-(4-fluorophenyl)-1-[(4-phenylpiperazin-1-yl)methyl]-4,5-dihydro-1*H*-1,2,4-triazole-5-thione ($C_{29}H_{34}FN_5S$) (I), and 3-(adamantan-1-yl)-4-(4-fluorophenyl)-1-{[4-(2-methoxyphenyl)piperazin-1-yl]-methyl}-4,5-dihydro-1*H*-1,2,4-triazole-5-thione ($C_{30}H_{36}FN_5OS$) (II), fluorophenyl, adamantane and piperazine moieties are linked to a planar triazole ring. There is an additional phenyl ring on the piperazine ring in (I) and a methoxyphenyl ring in (II). In compound (I), the fluorophenyl and phenyl rings are inclined to the triazole ring by 86.55 (13) and 60.52 (12)°, respectively, and the two aryl rings are inclined to one another by 66.37 (13)°. In compound (II), the corresponding dihedral angles are 83.35 (13), 71.38 (15) and 11.97 (16)°, respectively. The crystal structure of (I) shows pairs of C—H···F hydrogen bonds forming inversion dimers, while in the crystal of compound (II), in addition to the C—H···F hydrogen bonds that generate chains parallel to the *b* axis, there are C—H···π interactions present that link the chains to form layers parallel to the *ab* plane.

1. Chemical context

The incorporation of an adamantyl moiety into various bioactive molecules results in compounds with relatively high lipophilicity, which in turn modifies the bioavailability and modulates the therapeutic indices through various mechanisms (Liu *et al.*, 2011; Lamoureux & Artavia, 2010). Several adamantane-based drugs have been developed as antiviral (Davies *et al.*, 1964; Togo *et al.*, 1968; Rosenthal *et al.*, 1982; El-Emam *et al.*, 2004; Burstein *et al.*, 1999; Balzarini *et al.*, 2009), anti-Parkinsonian (Schwab *et al.*, 1969) and hypoglycaemic (Villhauer *et al.*, 2003; Augeri *et al.*, 2005) drugs. In addition, numerous adamantane-based analogues have promising anticancer (Sun *et al.*, 2002), bactericidal (Protopopova *et al.*, 2005; El-Emam *et al.*, 2013; Kadi *et al.*, 2010; Al-Abdullah *et al.*; 2014; Al-Deeb *et al.*, 2006) and fungicidal (Omar *et al.*, 2010) activities. In a continuation of our ongoing studies on the pharmacological and structural properties of adamantyl 1,2,4-triazole derivatives (Al-Abdullah *et al.*, 2012; Al-Tamimi *et al.*, 2014; El-Emam *et al.*; 2013; 2014), we report herein on the synthesis and crystal structures of the title adamantyl-substituted 1,2,4-triazole-5-thione *N*-Mannich bases, (I) and (II).



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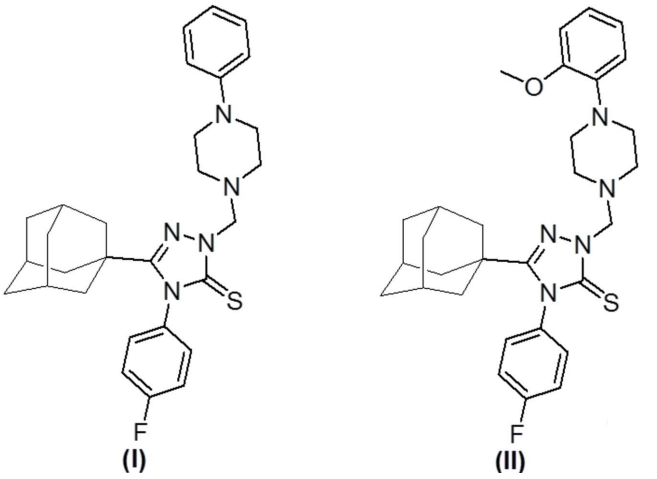

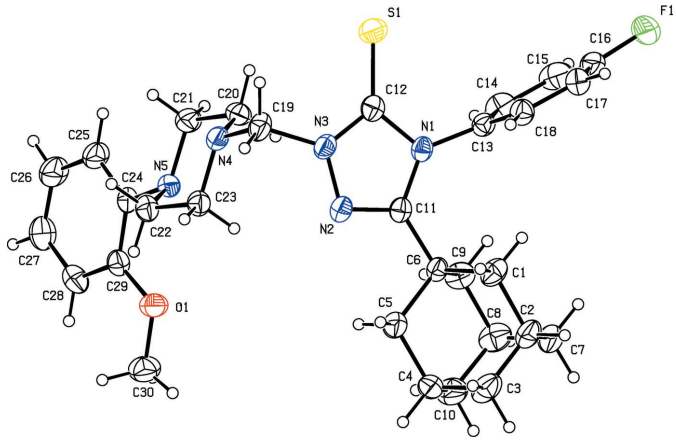

Figure 2
The molecular structure of compound (II), with the atom labelling and 30% probability displacement ellipsoids.

2. Structural commentary

The molecular structures of the title compounds, (I) and (II), are illustrated in Figs. 1 and 2, respectively. In both molecules the piperazine rings have a chair conformation, with the N-bound substituents occupying equatorial positions. In (I), the fluorophenyl ring (C13–C18) and the phenyl ring (C24–C29) are inclined to the triazole ring (N1–N3/C11/C12) by 86.55 (13) and 60.52 (12)°, respectively. The two aryl rings are inclined to one another by 66.37 (13)°. In compound (II), the fluorophenyl ring (C13–C18) and the phenyl ring (C24–C29) are inclined to the triazole ring (N1–N3/C11/C12) by 83.35 (13) and 71.38 (15)°, respectively, while the two aryl rings are inclined to one another by only 11.97 (16)°. This difference in conformation is illustrated by the structural overlap diagram, shown in Fig. 3.

3. Supramolecular features

In the crystal of compound (I), molecules are linked by pairs of C—H···F hydrogen bonds, forming inversion dimers (Table 1 and Fig. 4). In compound (II), molecules are linked by C—H···F hydrogen bonds, forming chains parallel to the *b*-axis direction. The chains are linked by C—H···π interactions, forming layers parallel to the *ab* plane (Table 2 and Fig. 5).

4. Database survey

A search of the Cambridge Structural Database (Version 5.38, last update May 2017; Groom *et al.*, 2016) for the 3-(adamantan-1-yl)-4-[(piperazin-1-yl) methyl]-1,2,4-triazole-3(3*H*)-thione moiety gave 14 hits. One compound, 3-(adamantan-1-yl)-4-phenyl-1-[(4-phenylpiperazin-1-yl)methyl]-1*H*-1,2,4-triazole-5(4*H*)-thione (GAPWUR; Al-Abdullah *et al.*, 2012), is very similar to compound (I). It has a phenyl ring substituent on the piperazine ring and a phenyl ring substituent on the triazole ring, which are inclined to one another by 72.4 (2)°, and by 89.0 (2) and 74.4 (2)°, respectively, to the triazole ring. In compound (I), the corresponding dihedral angles are 66.37 (13), 86.55 (13) and 60.52 (12)°, respectively. Two compounds have a 2-methoxyphenyl ring

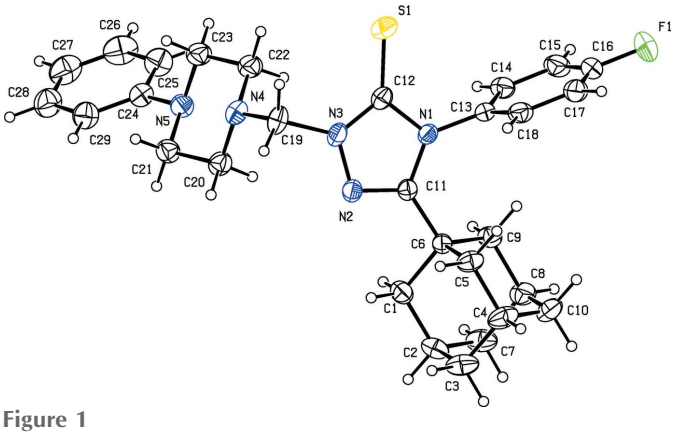

Figure 1
The molecular structure of compound (I), with the atom labelling and 30% probability displacement ellipsoids.

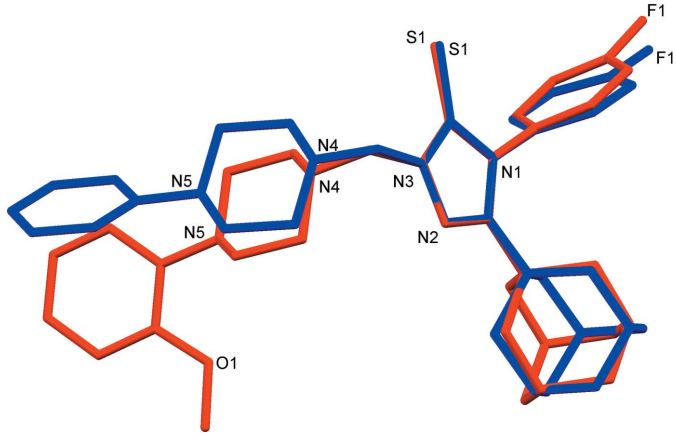

Figure 3
A structural overlap of compounds (I) and (II) [colour code: (I) blue, (II) red].

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

D—H···A	D—H	H···A	D···A	D—H···A
C22—H22B···F1^{i}	0.99	2.49	3.332 (4)	142

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

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

*Cg*1 and *Cg*8 are the centroids of rings (N1–N3/C11/C12) and (C24–C29), respectively.

D—H···A	D—H	H···A	D···A	D—H···A
C21—H21A···F1^{i}	0.97	2.47	3.407 (3)	162
C18—H18A···*Cg*1^{ii}	0.93	2.81	3.661	152
C9—H9A···*Cg*8^{iii}	0.97	2.80	3.697	155

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

substituent on the piperazine ring, *viz*. (3-(1-adamantyl)-1-{[4-(2-methoxyphenyl)piperazin-1-yl]methyl}-4-methyl-1*H*-1,2,4-triazole-5(4*H*)-thione (YUPVIP; El-Emam *et al.*, 2014), with a methyl substituent on the triazole ring, and 3-(adamantan-1-yl)-4-ethyl-1-{[4-(2-methoxyphenyl)piperazin-1-yl]methyl}-1*H*-1, 2,4-triazole-5(4*H*)-thione (RITMUE; Al-Tamimi *et al.*, 2010) with an ethyl substituent on the triazole ring. In these two compounds the methoxyphenyl rings are inclined to the triazole ring by 67.1 (1) and 59.4 (1)°, respectively. In compound (II), the corresponding dihedral angle is 71.38 (15)°.

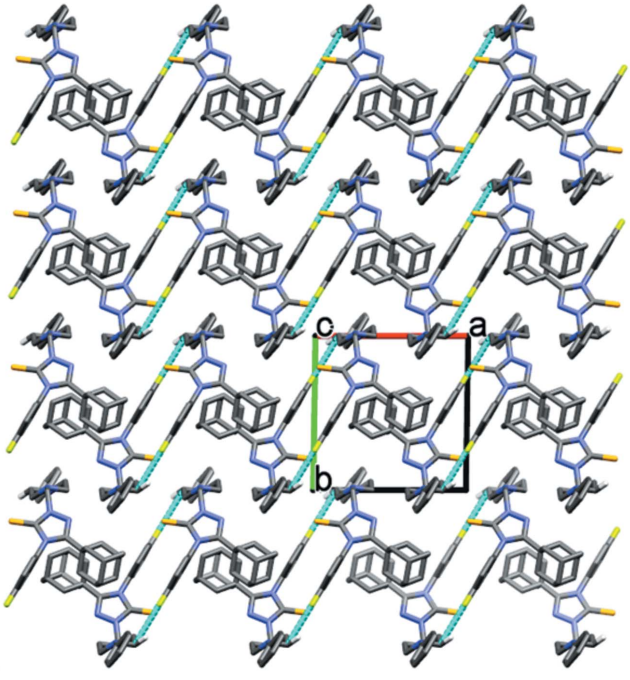

Figure 4
The crystal packing of compound (I), viewed along the *c* axis. The hydrogen bonds are shown as dashed lines (see Table 1), and only the H atoms involved in these interactions have been included.

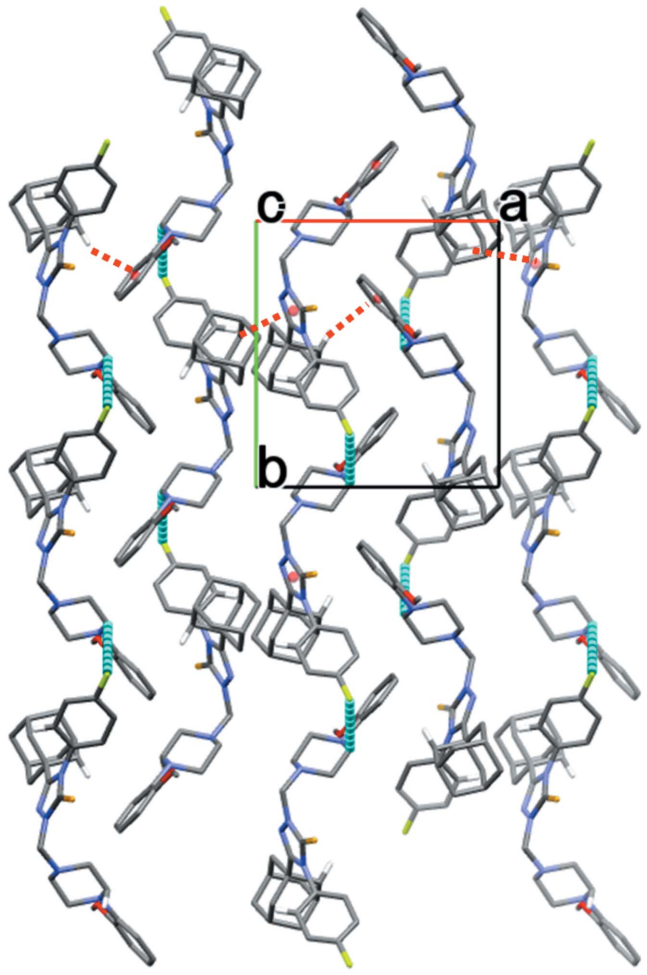

Figure 5
The crystal packing of compound (II), viewed along the *c* axis, showing the C—H···F hydrogen bonds (dashed cyan lines) and some of the C—H···π interactions (dashed red lines); see Table 2. Only the H atoms involved in these interactions have been included.

5. Synthesis and crystallization

The title compounds, (I) and (II), were synthesized *via* the reaction of 3-(adamantan-1-yl)-4-(4-fluoromethyl)-4*H*-1,2,4-triazole-5-thiol (Al-Deeb *et al.*, 2006) with the appropriate monosubstituted piperazie and a formaldehyde solution, as outlined in Fig. 6.

Compound (I): 1-Phenylpiperazine (325 mg, 2 mmol) and a 37% formaldehyde solution (1 ml) were added to a solution of 3-(adamantan-1-yl)-4-(4-fluoromethyl)-4*H*-1,2,4-triazole-5-thiol (659 mg, 2 mmol) in ethanol (10 ml), and the mixture was heated under reflux for 1 h then allowed to stand overnight. Cold water (3 ml) was slowly added and the mixture was stirred for 20 min. The precipitated crude product was filtered, washed with water, dried, and crystallized from ethanol to yield 846 mg (84%) of compound (I) as colourless plate-shaped crystals (m.p. 469–471 K). ^{1}H NMR (700.17 MHz): δ 1.47–1.49 (*m*, 3H, adamantane-H), 1.60–1.62 (*m*, 3H, adamantane-H), 1.80 (*s*, 6H, adamantane-H), 1.89 (*s*, 3H,

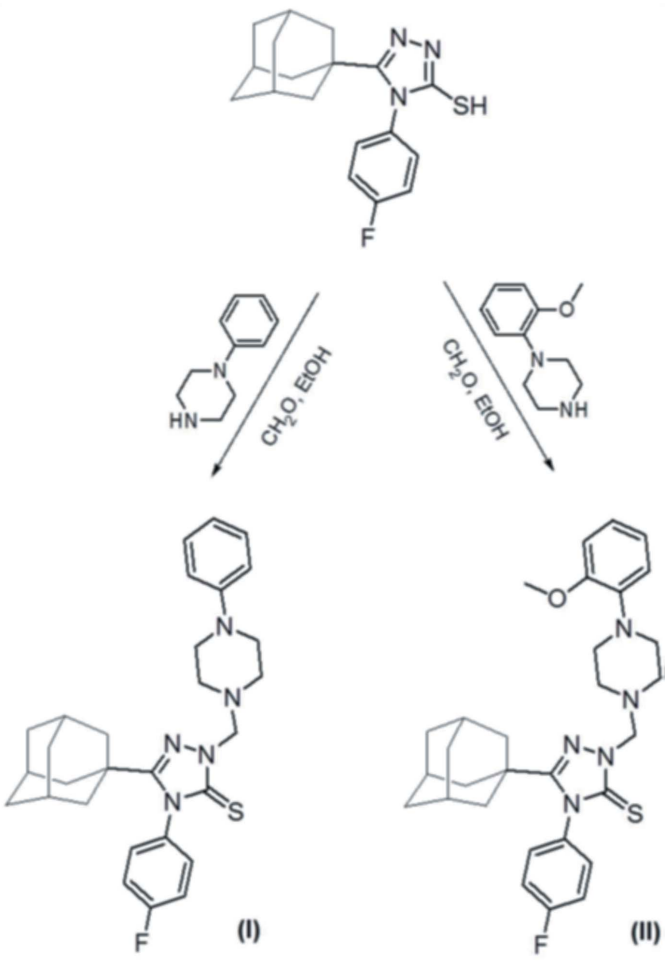

Figure 6
Reaction schemes for the syntheses of compounds (I) and (II).

adamantane-H), 2.89–2.91 (*m*, 4H, piperazine-H), 3.14–3.15 (*m*, 4H, piperazine-H), 5.14 (*s*, 2H, CH_2), 6.77–6.79 (*m*, 1H, Ar-H), 6.94 (*d*, 2H, Ar-H, J = 8.4 Hz), 7.20–7.22 (*m*, 2H, Ar-H), 7.41–7.49 (*m*, 4H, Ar-H). ^{13}C NMR (125.76 MHz): δ 27.61, 36.07, 39.62, 39.74 (adamantane-C), 48.73, 50.30 (piperazine-C), 69.06 (CH_2), 116.0, 116.78, 119.41, 129.37, 132.80, 133.10, 151.48, 156.28 (Ar-C), 162.17 (triazole C-3), 170.95 (C═S).

Compound (II): 1-(2-Methoxyphenyl)piperazine (385 mg, 2 mmol) and a 37% formaldehyde solution (1 ml) were added to a solution of 3-(adamantan-1-yl)-4-(4-fluoromethyl)-4*H*-1,2,4-triazole-5-thiol (659 mg, 2 mmol) in ethanol (10 ml), and the mixture was heated under reflux for 1 h then allowed to stand overnight. The precipitated crude product was filtered, washed with cold ethanol, dried, and crystallized from ethanol to yield 865 mg (81%) of compound (II) as colourless block-like crystals (m.p. 462–464 K). ^{1}H NMR (700.17 MHz): δ 1.49–1.50 (*m*, 3H, adamantane-H), 1.61–1.63 (*m*, 3H, adamantane-H), 1.83 (*s*, 6H, adamantane-H), 1.90 (*s*, 3H, adamantane-H), 2.89–2.90 (*m*, 4H, piperazine-H), 2.96–2.98 (*m*, 4H, piperazine-H), 3.78 (*s*, 3H, OCH_3), 5.11 (*s*, 2H, CH2), 6.88–6.96 (*m*, 4H, Ar-H), 7.42–7.52 (*m*, 4H, Ar-H). ^{13}C NMR (125.76 MHz): δ 27.61, 35.77, 36.07, 39.61 (adamantane-C), 50.54, 50.58 (piperazine-C), 55.65 (OCH_3), 69.39 (CH_2), 112.08, 116.78, 118.51, 121.23, 123.02, 132.79, 133.13, 141.53, 152.36, 156.32 (Ar-C), 162.17 (triazole C-3), 171.0 (C═S).

Suitable single crystals of compounds (I) and (II) were obtained by slow evaporation of $CHCl_3$:EtOH solutions (1:1, 5 ml) at room temperature.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3.The C-bound H atoms were positioned geometrically and treated as riding atoms: C—H = 0.93–1.00 Å with U_{iso}(H) = 1.5U_{eq}(C-methyl) and 1.2U_{eq}(C) for other H atoms.

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Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{29}H_{34}FN_5S$	$C_{30}H_{36}FN_5OS$
M_r	503.67	533.7
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/c$
Temperature (K)	296	293
a, b, c (Å)	10.4173 (5), 10.9849 (5), 12.0002 (6)	11.3074 (7), 12.1576 (8), 20.4976 (13)
α, β, γ (°)	72.769 (2), 84.623 (2), 89.244 (2)	90, 101.328 (2), 90
V (Å^3)	1305.66 (11)	2762.9 (3)
Z	2	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.16	0.16
Crystal size (mm)	0.45 × 0.33 × 0.09	0.42 × 0.19 × 0.16
Data collection		
Diffractometer	Bruker APEXII CCD	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.939, 0.986	0.965, 0.975
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	49580, 6011, 3533	67287, 6350, 3203
R_{int}	0.076	0.134
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650	0.649
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.061, 0.163, 1.03	0.055, 0.133, 1.01
No. of reflections	6007	6347
No. of parameters	325	344
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.25	0.16, −0.22

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXS2016* (Sheldrick, 2008), *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008), *SHELXL2016* (Sheldrick, 2015), *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

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supporting information

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Computing details

For both structures, data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXS2016* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2016* (Sheldrick, 2015), *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

3-(Adamantan-1-yl)-4-(4-fluorophenyl)-1-[(4-phenylpiperazin-1-yl)methyl]-4,5-dihydro-1*H*-1,2,4-triazole-5-thione (I)

Crystal data

$C_{29}H_{34}FN_5S$
$M_r = 503.67$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 10.4173$ (5) Å
$b = 10.9849$ (5) Å
$c = 12.0002$ (6) Å
$\alpha = 72.769$ (2)°
$\beta = 84.623$ (2)°
$\gamma = 89.244$ (2)°
$V = 1305.66$ (11) Å^3
$Z = 2$
$F(000) = 536$
$D_x = 1.281$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9217 reflections
$\theta = 2.5$–$23.7°$
$\mu = 0.16$ mm^{-1}
$T = 296$ K
Plate, colourless
0.45 × 0.33 × 0.09 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
$T_{min} = 0.939$, $T_{max} = 0.986$
49580 measured reflections
6011 independent reflections
3533 reflections with $I > 2\sigma(I)$
$R_{int} = 0.076$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.5°$
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.163$
$S = 1.03$
6007 reflections
325 parameters
0 restraints
0 constraints
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
$w = 1/[\sigma^2(F_o^2) + (0.065P)^2 + 0.6113P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ 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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.97289 (7)	0.79726 (7)	0.53684 (7)	0.0643 (2)
F1	0.9953 (2)	0.2584 (2)	0.8886 (2)	0.1173 (9)
N1	0.74459 (18)	0.67026 (17)	0.62307 (16)	0.0391 (5)
N2	0.59860 (19)	0.80635 (18)	0.54133 (17)	0.0454 (5)
N3	0.7211 (2)	0.85604 (18)	0.50475 (17)	0.0472 (5)
N4	0.7392 (2)	1.01606 (19)	0.30911 (17)	0.0494 (5)
N5	0.7233 (2)	0.9884 (2)	0.08319 (18)	0.0497 (5)
C1	0.3802 (2)	0.6521 (3)	0.6157 (2)	0.0528 (7)
H1A	0.3644	0.7428	0.6098	0.063*
H1B	0.3933	0.6444	0.5353	0.063*
C2	0.2634 (3)	0.5709 (3)	0.6825 (3)	0.0592 (7)
H2A	0.1852	0.6003	0.64	0.071*
C3	0.2423 (3)	0.5859 (3)	0.8050 (3)	0.0716 (9)
H3A	0.226	0.6764	0.7997	0.086*
H3B	0.1661	0.5346	0.8486	0.086*
C4	0.3614 (3)	0.5414 (3)	0.8693 (2)	0.0660 (8)
H4A	0.3472	0.5516	0.9496	0.079*
C5	0.4791 (2)	0.6218 (3)	0.8034 (2)	0.0514 (7)
H5A	0.5559	0.5932	0.8463	0.062*
H5B	0.4646	0.7123	0.7986	0.062*
C6	0.5025 (2)	0.6083 (2)	0.67938 (19)	0.0365 (5)
C7	0.2865 (3)	0.4317 (3)	0.6911 (3)	0.0669 (8)
H7A	0.3007	0.4216	0.6117	0.08*
H7B	0.2101	0.3795	0.7328	0.08*

C8	0.4048 (3)	0.3872 (3)	0.7577 (3)	0.0606 (8)
H8A	0.4197	0.2952	0.7647	0.073*
C9	0.5233 (2)	0.4665 (2)	0.6893 (2)	0.0487 (6)
H9A	0.5367	0.4557	0.61	0.058*
H9B	0.6011	0.4362	0.7303	0.058*
C10	0.3844 (3)	0.4035 (3)	0.8797 (3)	0.0727 (9)
H10A	0.3093	0.3513	0.9245	0.087*
H10B	0.4614	0.3744	0.9223	0.087*
C11	0.6145 (2)	0.6934 (2)	0.61309 (19)	0.0381 (5)
C12	0.8141 (2)	0.7769 (2)	0.5529 (2)	0.0442 (6)
C13	0.8086 (2)	0.5622 (2)	0.6933 (2)	0.0393 (5)
C14	0.8596 (2)	0.4721 (2)	0.6427 (2)	0.0505 (6)
H14A	0.8511	0.4817	0.5624	0.061*
C15	0.9223 (3)	0.3690 (3)	0.7086 (3)	0.0648 (8)
H15A	0.9572	0.3058	0.6753	0.078*
C16	0.9332 (3)	0.3597 (3)	0.8221 (3)	0.0700 (9)
C17	0.8867 (3)	0.4476 (3)	0.8746 (2)	0.0679 (9)
H17A	0.897	0.4375	0.9546	0.081*
C18	0.8236 (2)	0.5524 (3)	0.8079 (2)	0.0520 (7)
H18A	0.7913	0.6164	0.8412	0.062*
C19	0.7401 (3)	0.9915 (2)	0.4322 (2)	0.0548 (7)
H19A	0.6716	1.0425	0.459	0.066*
H19B	0.8236	1.0223	0.4476	0.066*
C20	0.6161 (3)	1.0001 (3)	0.2677 (2)	0.0530 (7)
H20A	0.5484	1.0444	0.3039	0.064*
H20B	0.5925	0.9084	0.2908	0.064*
C21	0.6245 (3)	1.0548 (3)	0.1353 (2)	0.0530 (7)
H21A	0.5403	1.0445	0.1069	0.064*
H21B	0.6462	1.1469	0.1121	0.064*
C23	0.8397 (3)	0.9514 (3)	0.2571 (2)	0.0574 (7)
H23A	0.8203	0.8588	0.2814	0.069*
H23B	0.9236	0.9644	0.2848	0.069*
C22	0.8475 (3)	1.0037 (3)	0.1250 (2)	0.0585 (7)
H22A	0.8722	1.0951	0.1006	0.07*
H22B	0.9145	0.958	0.0897	0.07*
C24	0.7295 (3)	1.0128 (2)	−0.0403 (2)	0.0502 (6)
C25	0.8004 (3)	0.9305 (3)	−0.0903 (3)	0.0678 (8)
H25A	0.8423	0.8599	−0.0417	0.081*
C26	0.8102 (4)	0.9509 (4)	−0.2097 (3)	0.0820 (10)
H26A	0.8584	0.8938	−0.2426	0.098*
C27	0.7515 (4)	1.0521 (4)	−0.2813 (3)	0.0837 (11)
H27A	0.7599	1.066	−0.3637	0.1*
C28	0.6804 (3)	1.1335 (4)	−0.2340 (3)	0.0782 (10)
H28A	0.6387	1.2036	−0.2835	0.094*
C29	0.6692 (3)	1.1139 (3)	−0.1138 (3)	0.0633 (8)
H29A	0.6195	1.1706	−0.0816	0.076*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0496 (4)	0.0660 (5)	0.0691 (5)	−0.0162 (3)	0.0024 (3)	−0.0092 (4)
F1	0.0819 (14)	0.1034 (16)	0.1216 (18)	0.0377 (12)	−0.0074 (12)	0.0334 (13)
N1	0.0401 (11)	0.0368 (11)	0.0371 (11)	−0.0016 (8)	0.0025 (8)	−0.0076 (8)
N2	0.0497 (12)	0.0399 (11)	0.0410 (11)	0.0049 (9)	0.0022 (9)	−0.0058 (9)
N3	0.0543 (13)	0.0385 (11)	0.0415 (12)	−0.0042 (10)	0.0042 (9)	−0.0030 (9)
N4	0.0572 (13)	0.0475 (12)	0.0411 (12)	−0.0079 (10)	−0.0001 (10)	−0.0107 (9)
N5	0.0502 (13)	0.0550 (13)	0.0439 (12)	0.0054 (10)	−0.0022 (9)	−0.0155 (10)
C1	0.0481 (15)	0.0545 (16)	0.0541 (16)	0.0109 (12)	−0.0079 (12)	−0.0130 (13)
C2	0.0377 (14)	0.0703 (19)	0.074 (2)	0.0088 (13)	−0.0132 (13)	−0.0255 (15)
C3	0.0447 (16)	0.090 (2)	0.085 (2)	−0.0052 (15)	0.0155 (15)	−0.0409 (18)
C4	0.0536 (17)	0.098 (2)	0.0467 (16)	−0.0167 (16)	0.0137 (13)	−0.0262 (16)
C5	0.0447 (14)	0.0666 (17)	0.0468 (15)	−0.0077 (12)	0.0044 (11)	−0.0248 (13)
C6	0.0380 (12)	0.0370 (12)	0.0341 (12)	0.0051 (10)	0.0005 (9)	−0.0114 (10)
C7	0.0460 (16)	0.075 (2)	0.089 (2)	−0.0099 (14)	−0.0048 (15)	−0.0379 (17)
C8	0.0475 (15)	0.0402 (15)	0.093 (2)	−0.0066 (12)	−0.0009 (14)	−0.0186 (14)
C9	0.0415 (14)	0.0434 (14)	0.0637 (17)	0.0020 (11)	−0.0014 (12)	−0.0206 (12)
C10	0.0544 (18)	0.081 (2)	0.064 (2)	−0.0212 (16)	0.0055 (14)	0.0049 (16)
C11	0.0438 (13)	0.0378 (13)	0.0320 (12)	0.0050 (10)	0.0003 (10)	−0.0107 (10)
C12	0.0507 (15)	0.0422 (14)	0.0381 (13)	−0.0036 (11)	0.0031 (11)	−0.0114 (11)
C13	0.0324 (12)	0.0402 (13)	0.0413 (14)	−0.0017 (10)	0.0008 (10)	−0.0073 (10)
C14	0.0423 (14)	0.0535 (16)	0.0567 (16)	0.0025 (12)	−0.0017 (12)	−0.0185 (13)
C15	0.0451 (16)	0.0540 (18)	0.092 (2)	0.0079 (13)	−0.0001 (15)	−0.0185 (16)
C16	0.0418 (16)	0.066 (2)	0.079 (2)	0.0101 (14)	0.0003 (15)	0.0128 (17)
C17	0.0473 (16)	0.099 (2)	0.0415 (16)	0.0024 (16)	−0.0036 (12)	0.0037 (16)
C18	0.0403 (14)	0.0688 (18)	0.0453 (15)	−0.0011 (12)	0.0010 (11)	−0.0160 (13)
C19	0.0785 (19)	0.0372 (14)	0.0445 (15)	−0.0067 (13)	−0.0011 (13)	−0.0069 (11)
C20	0.0562 (16)	0.0494 (15)	0.0514 (16)	0.0017 (12)	0.0045 (12)	−0.0148 (12)
C21	0.0516 (15)	0.0584 (16)	0.0490 (16)	0.0068 (13)	−0.0034 (12)	−0.0164 (13)
C23	0.0482 (15)	0.0715 (19)	0.0477 (16)	−0.0004 (13)	−0.0034 (12)	−0.0105 (13)
C22	0.0480 (16)	0.076 (2)	0.0475 (16)	0.0016 (14)	−0.0001 (12)	−0.0136 (14)
C24	0.0522 (15)	0.0520 (16)	0.0479 (15)	−0.0042 (12)	−0.0024 (12)	−0.0176 (12)
C25	0.076 (2)	0.069 (2)	0.063 (2)	0.0030 (16)	−0.0014 (16)	−0.0289 (16)
C26	0.084 (2)	0.103 (3)	0.073 (2)	−0.006 (2)	0.0066 (19)	−0.051 (2)
C27	0.086 (2)	0.117 (3)	0.0518 (19)	−0.022 (2)	−0.0013 (18)	−0.031 (2)
C28	0.079 (2)	0.094 (3)	0.056 (2)	−0.0078 (19)	−0.0103 (17)	−0.0124 (18)
C29	0.0690 (19)	0.0678 (19)	0.0543 (18)	0.0011 (15)	−0.0054 (14)	−0.0200 (15)

Geometric parameters (Å, º)

S1—C12	1.658 (3)	C8—H8A	1
F1—C16	1.358 (3)	C9—H9A	0.99
N1—C11	1.385 (3)	C9—H9B	0.99
N1—C12	1.389 (3)	C10—H10A	0.99
N1—C13	1.433 (3)	C10—H10B	0.99
N2—C11	1.303 (3)	C13—C18	1.371 (3)

N2—N3	1.375 (3)	C13—C14	1.383 (3)
N3—C12	1.346 (3)	C14—C15	1.373 (4)
N3—C19	1.490 (3)	C14—H14A	0.95
N4—C19	1.422 (3)	C15—C16	1.351 (5)
N4—C20	1.449 (3)	C15—H15A	0.95
N4—C23	1.452 (3)	C16—C17	1.361 (5)
N5—C24	1.421 (3)	C17—C18	1.389 (4)
N5—C21	1.453 (3)	C17—H17A	0.95
N5—C22	1.459 (3)	C18—H18A	0.95
C1—C2	1.530 (4)	C19—H19A	0.99
C1—C6	1.545 (3)	C19—H19B	0.99
C1—H1A	0.99	C20—C21	1.518 (3)
C1—H1B	0.99	C20—H20A	0.99
C2—C7	1.519 (4)	C20—H20B	0.99
C2—C3	1.523 (4)	C21—H21A	0.99
C2—H2A	1	C21—H21B	0.99
C3—C4	1.521 (4)	C23—C22	1.513 (4)
C3—H3A	0.99	C23—H23A	0.99
C3—H3B	0.99	C23—H23B	0.99
C4—C10	1.501 (4)	C22—H22A	0.99
C4—C5	1.529 (4)	C22—H22B	0.99
C4—H4A	1	C24—C29	1.383 (4)
C5—C6	1.536 (3)	C24—C25	1.394 (4)
C5—H5A	0.99	C25—C26	1.377 (4)
C5—H5B	0.99	C25—H25A	0.95
C6—C11	1.511 (3)	C26—C27	1.365 (5)
C6—C9	1.540 (3)	C26—H26A	0.95
C7—C8	1.528 (4)	C27—C28	1.370 (5)
C7—H7A	0.99	C27—H27A	0.95
C7—H7B	0.99	C28—C29	1.387 (4)
C8—C10	1.522 (4)	C28—H28A	0.95
C8—C9	1.539 (4)	C29—H29A	0.95

C11—N1—C12	108.39 (19)	N2—C11—N1	110.2 (2)
C11—N1—C13	130.57 (18)	N2—C11—C6	122.5 (2)
C12—N1—C13	121.01 (19)	N1—C11—C6	127.21 (19)
C11—N2—N3	105.12 (19)	N3—C12—N1	102.9 (2)
C12—N3—N2	113.44 (18)	N3—C12—S1	130.23 (18)
C12—N3—C19	125.9 (2)	N1—C12—S1	126.90 (19)
N2—N3—C19	120.1 (2)	C18—C13—C14	120.9 (2)
C19—N4—C20	116.2 (2)	C18—C13—N1	119.8 (2)
C19—N4—C23	114.0 (2)	C14—C13—N1	119.3 (2)
C20—N4—C23	110.5 (2)	C15—C14—C13	119.9 (3)
C24—N5—C21	116.6 (2)	C15—C14—H14A	120.1
C24—N5—C22	113.3 (2)	C13—C14—H14A	120.1
C21—N5—C22	109.8 (2)	C16—C15—C14	118.1 (3)
C2—C1—C6	110.4 (2)	C16—C15—H15A	120.9
C2—C1—H1A	109.6	C14—C15—H15A	120.9

C6—C1—H1A	109.6	C15—C16—F1	118.9 (3)
C2—C1—H1B	109.6	C15—C16—C17	123.8 (3)
C6—C1—H1B	109.6	F1—C16—C17	117.3 (3)
H1A—C1—H1B	108.1	C16—C17—C18	118.2 (3)
C7—C2—C3	109.6 (3)	C16—C17—H17A	120.9
C7—C2—C1	109.9 (2)	C18—C17—H17A	120.9
C3—C2—C1	109.2 (2)	C13—C18—C17	119.1 (3)
C7—C2—H2A	109.4	C13—C18—H18A	120.5
C3—C2—H2A	109.4	C17—C18—H18A	120.5
C1—C2—H2A	109.4	N4—C19—N3	116.3 (2)
C4—C3—C2	109.3 (2)	N4—C19—H19A	108.2
C4—C3—H3A	109.8	N3—C19—H19A	108.2
C2—C3—H3A	109.8	N4—C19—H19B	108.2
C4—C3—H3B	109.8	N3—C19—H19B	108.2
C2—C3—H3B	109.8	H19A—C19—H19B	107.4
H3A—C3—H3B	108.3	N4—C20—C21	109.7 (2)
C10—C4—C3	109.9 (3)	N4—C20—H20A	109.7
C10—C4—C5	109.9 (2)	C21—C20—H20A	109.7
C3—C4—C5	110.1 (3)	N4—C20—H20B	109.7
C10—C4—H4A	109	C21—C20—H20B	109.7
C3—C4—H4A	109	H20A—C20—H20B	108.2
C5—C4—H4A	109	N5—C21—C20	109.2 (2)
C4—C5—C6	110.0 (2)	N5—C21—H21A	109.8
C4—C5—H5A	109.7	C20—C21—H21A	109.8
C6—C5—H5A	109.7	N5—C21—H21B	109.8
C4—C5—H5B	109.7	C20—C21—H21B	109.8
C6—C5—H5B	109.7	H21A—C21—H21B	108.3
H5A—C5—H5B	108.2	N4—C23—C22	109.7 (2)
C11—C6—C5	109.46 (19)	N4—C23—H23A	109.7
C11—C6—C9	113.41 (18)	C22—C23—H23A	109.7
C5—C6—C9	108.6 (2)	N4—C23—H23B	109.7
C11—C6—C1	108.72 (18)	C22—C23—H23B	109.7
C5—C6—C1	108.15 (19)	H23A—C23—H23B	108.2
C9—C6—C1	108.4 (2)	N5—C22—C23	110.3 (2)
C2—C7—C8	108.9 (2)	N5—C22—H22A	109.6
C2—C7—H7A	109.9	C23—C22—H22A	109.6
C8—C7—H7A	109.9	N5—C22—H22B	109.6
C2—C7—H7B	109.9	C23—C22—H22B	109.6
C8—C7—H7B	109.9	H22A—C22—H22B	108.1
H7A—C7—H7B	108.3	C29—C24—C25	118.1 (3)
C10—C8—C7	110.3 (2)	C29—C24—N5	123.6 (2)
C10—C8—C9	109.7 (2)	C25—C24—N5	118.3 (2)
C7—C8—C9	109.1 (2)	C26—C25—C24	120.4 (3)
C10—C8—H8A	109.2	C26—C25—H25A	119.8
C7—C8—H8A	109.2	C24—C25—H25A	119.8
C9—C8—H8A	109.2	C27—C26—C25	120.8 (3)
C8—C9—C6	109.8 (2)	C27—C26—H26A	119.6
C8—C9—H9A	109.7	C25—C26—H26A	119.6

C6—C9—H9A	109.7	C26—C27—C28	119.8 (3)
C8—C9—H9B	109.7	C26—C27—H27A	120.1
C6—C9—H9B	109.7	C28—C27—H27A	120.1
H9A—C9—H9B	108.2	C27—C28—C29	120.1 (3)
C4—C10—C8	109.3 (2)	C27—C28—H28A	120
C4—C10—H10A	109.8	C29—C28—H28A	120
C8—C10—H10A	109.8	C24—C29—C28	120.8 (3)
C4—C10—H10B	109.8	C24—C29—H29A	119.6
C8—C10—H10B	109.8	C28—C29—H29A	119.6
H10A—C10—H10B	108.3		

C11—N2—N3—C12	−0.5 (3)	C11—N1—C12—N3	−0.6 (2)
C11—N2—N3—C19	−172.6 (2)	C13—N1—C12—N3	−178.6 (2)
C6—C1—C2—C7	−59.7 (3)	C11—N1—C12—S1	178.75 (18)
C6—C1—C2—C3	60.6 (3)	C13—N1—C12—S1	0.8 (3)
C7—C2—C3—C4	60.3 (3)	C11—N1—C13—C18	−83.5 (3)
C1—C2—C3—C4	−60.2 (3)	C12—N1—C13—C18	94.0 (3)
C2—C3—C4—C10	−60.7 (3)	C11—N1—C13—C14	99.5 (3)
C2—C3—C4—C5	60.4 (3)	C12—N1—C13—C14	−83.1 (3)
C10—C4—C5—C6	60.9 (3)	C18—C13—C14—C15	2.3 (4)
C3—C4—C5—C6	−60.2 (3)	N1—C13—C14—C15	179.3 (2)
C4—C5—C6—C11	177.0 (2)	C13—C14—C15—C16	−0.6 (4)
C4—C5—C6—C9	−58.7 (3)	C14—C15—C16—F1	−179.9 (3)
C4—C5—C6—C1	58.7 (3)	C14—C15—C16—C17	−0.8 (5)
C2—C1—C6—C11	−178.1 (2)	C15—C16—C17—C18	0.5 (5)
C2—C1—C6—C5	−59.4 (3)	F1—C16—C17—C18	179.6 (2)
C2—C1—C6—C9	58.2 (3)	C14—C13—C18—C17	−2.5 (4)
C3—C2—C7—C8	−59.2 (3)	N1—C13—C18—C17	−179.5 (2)
C1—C2—C7—C8	60.8 (3)	C16—C17—C18—C13	1.2 (4)
C2—C7—C8—C10	59.0 (3)	C20—N4—C19—N3	69.5 (3)
C2—C7—C8—C9	−61.5 (3)	C23—N4—C19—N3	−60.7 (3)
C10—C8—C9—C6	−59.5 (3)	C12—N3—C19—N4	102.6 (3)
C7—C8—C9—C6	61.4 (3)	N2—N3—C19—N4	−86.3 (3)
C11—C6—C9—C8	−179.9 (2)	C19—N4—C20—C21	168.6 (2)
C5—C6—C9—C8	58.2 (3)	C23—N4—C20—C21	−59.5 (3)
C1—C6—C9—C8	−59.1 (3)	C24—N5—C21—C20	169.9 (2)
C3—C4—C10—C8	60.1 (3)	C22—N5—C21—C20	−59.5 (3)
C5—C4—C10—C8	−61.2 (3)	N4—C20—C21—N5	59.9 (3)
C7—C8—C10—C4	−59.6 (3)	C19—N4—C23—C22	−168.7 (2)
C9—C8—C10—C4	60.6 (3)	C20—N4—C23—C22	58.3 (3)
N3—N2—C11—N1	0.1 (2)	C24—N5—C22—C23	−168.7 (2)
N3—N2—C11—C6	176.33 (19)	C21—N5—C22—C23	59.0 (3)
C12—N1—C11—N2	0.3 (3)	N4—C23—C22—N5	−57.8 (3)
C13—N1—C11—N2	178.0 (2)	C21—N5—C24—C29	15.0 (4)
C12—N1—C11—C6	−175.7 (2)	C22—N5—C24—C29	−113.9 (3)
C13—N1—C11—C6	2.0 (4)	C21—N5—C24—C25	−165.1 (3)
C5—C6—C11—N2	−97.1 (3)	C22—N5—C24—C25	66.0 (3)
C9—C6—C11—N2	141.4 (2)	C29—C24—C25—C26	0.5 (4)

C1—C6—C11—N2	20.8 (3)	N5—C24—C25—C26	−179.4 (3)
C5—C6—C11—N1	78.4 (3)	C24—C25—C26—C27	0.4 (5)
C9—C6—C11—N1	−43.0 (3)	C25—C26—C27—C28	−1.0 (5)
C1—C6—C11—N1	−163.6 (2)	C26—C27—C28—C29	0.7 (5)
N2—N3—C12—N1	0.7 (3)	C25—C24—C29—C28	−0.8 (4)
C19—N3—C12—N1	172.3 (2)	N5—C24—C29—C28	179.1 (3)
N2—N3—C12—S1	−178.63 (18)	C27—C28—C29—C24	0.2 (5)
C19—N3—C12—S1	−7.1 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C22—H22*B*···F1^{i}	0.99	2.49	3.332 (4)	142

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

3-(Adamantan-1-yl)-4-(4-fluorophenyl)-1-{[4-(2-methoxyphenyl)piperazin-1-yl]methyl}-4,5-dihydro-1*H*-1,2,4-triazole-5-thione (II)

Crystal data

$C_{30}H_{36}FN_5OS$
M_r = 533.7
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 11.3074 (7) Å
b = 12.1576 (8) Å
c = 20.4976 (13) Å
β = 101.328 (2)°
V = 2762.9 (3) Å^3
Z = 4
$F(000)$ = 1136
D_x = 1.283 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8790 reflections
θ = 2.5–22.8°
μ = 0.16 mm^{-1}
T = 293 K
Block, colourless
0.42 × 0.19 × 0.16 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
T_{min} = 0.965, T_{max} = 0.975
67287 measured reflections
6350 independent reflections
3203 reflections with $I > 2\sigma(I)$
R_{int} = 0.134
θ_{max} = 27.5°, θ_{min} = 2.5°
h = −14→14
k = −15→15
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.133
S = 1.01
6347 reflections
344 parameters
0 restraints
0 constraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 0.8222P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.24167 (6)	0.32667 (6)	0.43345 (3)	0.0608 (2)
F1	0.3856 (2)	0.79567 (15)	0.45842 (10)	0.1199 (8)
O1	0.34615 (16)	−0.06806 (14)	0.77358 (8)	0.0618 (5)
N1	0.19559 (16)	0.42142 (14)	0.54744 (10)	0.0428 (5)
N3	0.13241 (17)	0.25598 (15)	0.53226 (9)	0.0469 (5)
N2	0.10390 (17)	0.28893 (15)	0.59193 (10)	0.0481 (5)
N4	0.18293 (18)	0.05911 (15)	0.54290 (9)	0.0476 (5)
N5	0.35533 (17)	−0.04464 (14)	0.64472 (9)	0.0439 (5)
C1	0.0665 (2)	0.5658 (2)	0.64261 (13)	0.0583 (7)
H1A	0.1087	0.6099	0.615	0.07*
H1B	−0.0137	0.5502	0.6174	0.07*
C2	0.0572 (3)	0.6293 (2)	0.70571 (14)	0.0645 (8)
H2A	0.014	0.6984	0.6932	0.077*
C3	−0.0100 (3)	0.5635 (2)	0.74851 (16)	0.0755 (9)
H3A	−0.0907	0.5476	0.7242	0.091*
H3B	−0.0167	0.6053	0.7879	0.091*
C4	0.0574 (3)	0.4566 (2)	0.76852 (15)	0.0750 (9)
H4A	0.0135	0.4131	0.7962	0.09*
C5	0.0666 (3)	0.3909 (2)	0.70539 (14)	0.0680 (8)
H5A	0.1084	0.3221	0.7179	0.082*
H5B	−0.0137	0.3739	0.6808	0.082*
C6	0.13488 (19)	0.45742 (17)	0.66124 (11)	0.0412 (6)
C7	0.1822 (3)	0.6551 (2)	0.74427 (15)	0.0741 (9)
H7A	0.2254	0.6987	0.717	0.089*
H7B	0.1765	0.6975	0.7836	0.089*
C8	0.2498 (2)	0.5495 (2)	0.76445 (14)	0.0688 (8)
H8A	0.3305	0.5668	0.7897	0.083*
C9	0.2610 (2)	0.4844 (2)	0.70174 (13)	0.0596 (7)
H9A	0.3051	0.4168	0.7143	0.072*
H9B	0.3054	0.5274	0.6748	0.072*
C10	0.1828 (3)	0.4810 (3)	0.80713 (14)	0.0807 (9)
H10A	0.2258	0.4127	0.8194	0.097*
H10B	0.1779	0.5207	0.8476	0.097*
C11	0.14343 (19)	0.39012 (18)	0.60057 (11)	0.0413 (6)
C12	0.1899 (2)	0.33355 (19)	0.50378 (12)	0.0455 (6)

C13	0.2440 (2)	0.52480 (18)	0.53089 (11)	0.0429 (6)
C14	0.3666 (2)	0.5415 (2)	0.54450 (13)	0.0610 (7)
H14A	0.4173	0.4902	0.5695	0.073*
C15	0.4142 (3)	0.6341 (3)	0.52107 (16)	0.0785 (10)
H15A	0.4969	0.6466	0.53	0.094*
C16	0.3373 (4)	0.7071 (2)	0.48460 (16)	0.0740 (9)
C17	0.2163 (3)	0.6944 (2)	0.47141 (14)	0.0666 (8)
H17A	0.1662	0.7472	0.4474	0.08*
C18	0.1692 (2)	0.60109 (19)	0.49462 (12)	0.0514 (6)
H18A	0.0862	0.5897	0.4857	0.062*
C19	0.1079 (2)	0.14220 (19)	0.50729 (12)	0.0550 (7)
H19A	0.0246	0.1246	0.5083	0.066*
H19B	0.1165	0.1399	0.4612	0.066*
C20	0.3118 (2)	0.0819 (2)	0.55064 (12)	0.0541 (7)
H20A	0.3322	0.146	0.5788	0.065*
H20B	0.3305	0.0983	0.5075	0.065*
C21	0.3869 (2)	−0.0149 (2)	0.58108 (12)	0.0560 (7)
H21A	0.3728	−0.0772	0.551	0.067*
H21B	0.4719	0.0039	0.588	0.067*
C22	0.2259 (2)	−0.06913 (19)	0.63489 (12)	0.0505 (6)
H22A	0.2053	−0.0894	0.677	0.061*
H22B	0.2066	−0.1307	0.6046	0.061*
C23	0.1536 (2)	0.03040 (19)	0.60675 (12)	0.0483 (6)
H23A	0.0681	0.0145	0.6011	0.058*
H23B	0.1721	0.0918	0.6373	0.058*
C24	0.4291 (2)	−0.12587 (18)	0.68294 (12)	0.0453 (6)
C25	0.5089 (2)	−0.1917 (2)	0.65777 (13)	0.0592 (7)
H25A	0.5185	−0.1814	0.6142	0.071*
C26	0.5751 (3)	−0.2730 (2)	0.69618 (16)	0.0722 (8)
H26A	0.6289	−0.316	0.6783	0.087*
C27	0.5616 (2)	−0.2903 (2)	0.76022 (16)	0.0686 (8)
H27A	0.6031	−0.3471	0.7852	0.082*
C28	0.4857 (2)	−0.2226 (2)	0.78764 (13)	0.0591 (7)
H28A	0.4781	−0.2326	0.8316	0.071*
C29	0.4211 (2)	−0.14002 (19)	0.74968 (13)	0.0482 (6)
C30	0.3295 (3)	−0.0813 (2)	0.83902 (13)	0.0715 (8)
H30A	0.2762	−0.0251	0.8491	0.107*
H30B	0.4059	−0.0755	0.8691	0.107*
H30C	0.295	−0.1523	0.8437	0.107*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0707 (5)	0.0622 (4)	0.0515 (4)	0.0101 (4)	0.0172 (3)	−0.0018 (3)
F1	0.190 (2)	0.0819 (13)	0.1131 (15)	−0.0717 (13)	0.0917 (15)	−0.0292 (11)
O1	0.0783 (13)	0.0624 (12)	0.0492 (11)	0.0115 (10)	0.0236 (9)	0.0040 (9)
N1	0.0404 (11)	0.0354 (11)	0.0527 (12)	0.0050 (9)	0.0089 (9)	−0.0016 (10)
N3	0.0552 (12)	0.0383 (11)	0.0467 (12)	0.0066 (10)	0.0083 (10)	−0.0046 (10)

N2	0.0510 (12)	0.0386 (12)	0.0548 (13)	0.0025 (9)	0.0107 (10)	−0.0056 (10)
N4	0.0623 (14)	0.0379 (11)	0.0429 (12)	0.0048 (10)	0.0111 (10)	−0.0022 (9)
N5	0.0504 (12)	0.0391 (11)	0.0444 (12)	0.0031 (9)	0.0144 (9)	0.0027 (9)
C1	0.0616 (17)	0.0520 (16)	0.0596 (17)	0.0177 (13)	0.0078 (13)	−0.0084 (13)
C2	0.078 (2)	0.0475 (15)	0.0713 (19)	0.0150 (14)	0.0220 (17)	−0.0104 (14)
C3	0.0684 (19)	0.074 (2)	0.093 (2)	−0.0041 (16)	0.0361 (17)	−0.0302 (17)
C4	0.104 (2)	0.0573 (18)	0.080 (2)	−0.0273 (17)	0.059 (2)	−0.0141 (16)
C5	0.087 (2)	0.0501 (16)	0.077 (2)	−0.0167 (14)	0.0416 (17)	−0.0140 (14)
C6	0.0379 (13)	0.0357 (13)	0.0507 (14)	0.0002 (10)	0.0109 (11)	−0.0047 (11)
C7	0.100 (2)	0.0554 (18)	0.073 (2)	−0.0244 (17)	0.0324 (18)	−0.0197 (15)
C8	0.0542 (17)	0.088 (2)	0.0603 (18)	−0.0096 (16)	0.0028 (14)	−0.0189 (16)
C9	0.0476 (15)	0.0727 (18)	0.0578 (17)	0.0027 (13)	0.0086 (13)	−0.0064 (14)
C10	0.115 (3)	0.075 (2)	0.0538 (18)	0.0137 (19)	0.0211 (19)	0.0017 (16)
C11	0.0348 (12)	0.0383 (14)	0.0496 (15)	0.0037 (10)	0.0055 (11)	−0.0008 (11)
C12	0.0408 (13)	0.0425 (14)	0.0509 (15)	0.0101 (11)	0.0033 (12)	0.0030 (12)
C13	0.0398 (14)	0.0430 (14)	0.0469 (14)	−0.0009 (11)	0.0110 (11)	−0.0053 (11)
C14	0.0442 (16)	0.0736 (19)	0.0639 (18)	−0.0019 (14)	0.0076 (13)	−0.0123 (15)
C15	0.062 (2)	0.101 (3)	0.078 (2)	−0.037 (2)	0.0290 (18)	−0.036 (2)
C16	0.108 (3)	0.0583 (19)	0.068 (2)	−0.038 (2)	0.049 (2)	−0.0253 (16)
C17	0.095 (2)	0.0471 (17)	0.0617 (19)	−0.0013 (16)	0.0260 (17)	0.0011 (14)
C18	0.0515 (15)	0.0464 (15)	0.0565 (16)	0.0025 (12)	0.0112 (13)	−0.0004 (13)
C19	0.0658 (17)	0.0420 (15)	0.0519 (16)	0.0003 (12)	−0.0017 (13)	−0.0088 (12)
C20	0.0679 (18)	0.0503 (15)	0.0493 (16)	0.0090 (13)	0.0246 (13)	0.0092 (12)
C21	0.0662 (17)	0.0518 (16)	0.0546 (16)	0.0119 (13)	0.0231 (14)	0.0034 (13)
C22	0.0526 (16)	0.0437 (14)	0.0548 (16)	−0.0053 (12)	0.0095 (12)	0.0005 (12)
C23	0.0492 (14)	0.0427 (14)	0.0539 (16)	−0.0028 (11)	0.0123 (12)	−0.0019 (12)
C24	0.0510 (14)	0.0332 (12)	0.0514 (16)	−0.0025 (11)	0.0093 (12)	−0.0007 (11)
C25	0.0704 (18)	0.0520 (16)	0.0562 (17)	0.0172 (14)	0.0147 (14)	0.0009 (13)
C26	0.077 (2)	0.0632 (19)	0.078 (2)	0.0246 (16)	0.0208 (17)	0.0007 (17)
C27	0.0634 (18)	0.0545 (17)	0.083 (2)	0.0115 (14)	0.0028 (16)	0.0106 (16)
C28	0.0621 (17)	0.0562 (16)	0.0572 (17)	−0.0057 (14)	0.0073 (14)	0.0131 (14)
C29	0.0474 (15)	0.0418 (14)	0.0559 (17)	−0.0045 (11)	0.0113 (13)	0.0020 (12)
C30	0.083 (2)	0.084 (2)	0.0484 (17)	−0.0038 (16)	0.0137 (15)	−0.0073 (15)

Geometric parameters (Å, º)

S1—C12	1.661 (2)	C8—H8A	0.98
F1—C16	1.364 (3)	C9—H9A	0.97
O1—C29	1.374 (3)	C9—H9B	0.97
O1—C30	1.400 (3)	C10—H10A	0.97
N1—C12	1.387 (3)	C10—H10B	0.97
N1—C11	1.390 (3)	C13—C18	1.372 (3)
N1—C13	1.438 (3)	C13—C14	1.374 (3)
N3—C12	1.342 (3)	C14—C15	1.375 (4)
N3—N2	1.384 (3)	C14—H14A	0.93
N3—C19	1.482 (3)	C15—C16	1.358 (4)
N2—C11	1.309 (3)	C15—H15A	0.93
N4—C19	1.425 (3)	C16—C17	1.351 (4)

N4—C23	1.454 (3)	C17—C18	1.377 (3)
N4—C20	1.461 (3)	C17—H17A	0.93
N5—C24	1.424 (3)	C18—H18A	0.93
N5—C21	1.464 (3)	C19—H19A	0.97
N5—C22	1.468 (3)	C19—H19B	0.97
C1—C2	1.528 (3)	C20—C21	1.512 (3)
C1—C6	1.537 (3)	C20—H20A	0.97
C1—H1A	0.97	C20—H20B	0.97
C1—H1B	0.97	C21—H21A	0.97
C2—C3	1.500 (4)	C21—H21B	0.97
C2—C7	1.510 (4)	C22—C23	1.510 (3)
C2—H2A	0.98	C22—H22A	0.97
C3—C4	1.522 (4)	C22—H22B	0.97
C3—H3A	0.97	C23—H23A	0.97
C3—H3B	0.97	C23—H23B	0.97
C4—C10	1.511 (4)	C24—C25	1.380 (3)
C4—C5	1.541 (4)	C24—C29	1.399 (3)
C4—H4A	0.98	C25—C26	1.388 (3)
C5—C6	1.531 (3)	C25—H25A	0.93
C5—H5A	0.97	C26—C27	1.367 (4)
C5—H5B	0.97	C26—H26A	0.93
C6—C11	1.507 (3)	C27—C28	1.385 (4)
C6—C9	1.538 (3)	C27—H27A	0.93
C7—C8	1.509 (4)	C28—C29	1.387 (3)
C7—H7A	0.97	C28—H28A	0.93
C7—H7B	0.97	C30—H30A	0.96
C8—C10	1.514 (4)	C30—H30B	0.96
C8—C9	1.536 (3)	C30—H30C	0.96

C29—O1—C30	118.5 (2)	N1—C11—C6	127.1 (2)
C12—N1—C11	108.77 (18)	N3—C12—N1	103.03 (19)
C12—N1—C13	119.51 (19)	N3—C12—S1	128.71 (18)
C11—N1—C13	131.62 (19)	N1—C12—S1	128.26 (18)
C12—N3—N2	113.44 (18)	C18—C13—C14	120.0 (2)
C12—N3—C19	125.4 (2)	C18—C13—N1	119.7 (2)
N2—N3—C19	121.01 (19)	C14—C13—N1	119.8 (2)
C11—N2—N3	105.01 (19)	C13—C14—C15	119.9 (3)
C19—N4—C23	114.40 (19)	C13—C14—H14A	120.1
C19—N4—C20	113.73 (19)	C15—C14—H14A	120.1
C23—N4—C20	110.29 (18)	C16—C15—C14	118.4 (3)
C24—N5—C21	116.26 (18)	C16—C15—H15A	120.8
C24—N5—C22	113.26 (18)	C14—C15—H15A	120.8
C21—N5—C22	109.82 (19)	C17—C16—C15	123.3 (3)
C2—C1—C6	109.8 (2)	C17—C16—F1	118.6 (4)
C2—C1—H1A	109.7	C15—C16—F1	118.0 (3)
C6—C1—H1A	109.7	C16—C17—C18	118.0 (3)
C2—C1—H1B	109.7	C16—C17—H17A	121
C6—C1—H1B	109.7	C18—C17—H17A	121

H1A—C1—H1B	108.2	C13—C18—C17	120.3 (3)
C3—C2—C7	109.5 (2)	C13—C18—H18A	119.8
C3—C2—C1	110.6 (2)	C17—C18—H18A	119.8
C7—C2—C1	109.6 (2)	N4—C19—N3	115.62 (19)
C3—C2—H2A	109	N4—C19—H19A	108.4
C7—C2—H2A	109	N3—C19—H19A	108.4
C1—C2—H2A	109	N4—C19—H19B	108.4
C2—C3—C4	109.2 (2)	N3—C19—H19B	108.4
C2—C3—H3A	109.8	H19A—C19—H19B	107.4
C4—C3—H3A	109.8	N4—C20—C21	111.4 (2)
C2—C3—H3B	109.8	N4—C20—H20A	109.4
C4—C3—H3B	109.8	C21—C20—H20A	109.4
H3A—C3—H3B	108.3	N4—C20—H20B	109.4
C10—C4—C3	110.0 (2)	C21—C20—H20B	109.4
C10—C4—C5	109.3 (2)	H20A—C20—H20B	108
C3—C4—C5	109.2 (3)	N5—C21—C20	110.12 (19)
C10—C4—H4A	109.4	N5—C21—H21A	109.6
C3—C4—H4A	109.4	C20—C21—H21A	109.6
C5—C4—H4A	109.4	N5—C21—H21B	109.6
C6—C5—C4	110.3 (2)	C20—C21—H21B	109.6
C6—C5—H5A	109.6	H21A—C21—H21B	108.2
C4—C5—H5A	109.6	N5—C22—C23	109.88 (18)
C6—C5—H5B	109.6	N5—C22—H22A	109.7
C4—C5—H5B	109.6	C23—C22—H22A	109.7
H5A—C5—H5B	108.1	N5—C22—H22B	109.7
C11—C6—C5	108.80 (19)	C23—C22—H22B	109.7
C11—C6—C1	111.76 (19)	H22A—C22—H22B	108.2
C5—C6—C1	108.2 (2)	N4—C23—C22	109.52 (19)
C11—C6—C9	110.99 (18)	N4—C23—H23A	109.8
C5—C6—C9	108.4 (2)	C22—C23—H23A	109.8
C1—C6—C9	108.60 (19)	N4—C23—H23B	109.8
C8—C7—C2	109.8 (2)	C22—C23—H23B	109.8
C8—C7—H7A	109.7	H23A—C23—H23B	108.2
C2—C7—H7A	109.7	C25—C24—C29	117.8 (2)
C8—C7—H7B	109.7	C25—C24—N5	123.5 (2)
C2—C7—H7B	109.7	C29—C24—N5	118.7 (2)
H7A—C7—H7B	108.2	C24—C25—C26	121.4 (3)
C7—C8—C10	110.0 (2)	C24—C25—H25A	119.3
C7—C8—C9	109.2 (2)	C26—C25—H25A	119.3
C10—C8—C9	109.4 (2)	C27—C26—C25	120.3 (3)
C7—C8—H8A	109.4	C27—C26—H26A	119.9
C10—C8—H8A	109.4	C25—C26—H26A	119.9
C9—C8—H8A	109.4	C26—C27—C28	119.5 (3)
C8—C9—C6	110.0 (2)	C26—C27—H27A	120.2
C8—C9—H9A	109.7	C28—C27—H27A	120.2
C6—C9—H9A	109.7	C27—C28—C29	120.2 (3)
C8—C9—H9B	109.7	C27—C28—H28A	119.9
C6—C9—H9B	109.7	C29—C28—H28A	119.9

H9A—C9—H9B	108.2	O1—C29—C28	123.7 (2)
C4—C10—C8	109.2 (2)	O1—C29—C24	115.7 (2)
C4—C10—H10A	109.8	C28—C29—C24	120.6 (2)
C8—C10—H10A	109.8	O1—C30—H30A	109.5
C4—C10—H10B	109.8	O1—C30—H30B	109.5
C8—C10—H10B	109.8	H30A—C30—H30B	109.5
H10A—C10—H10B	108.3	O1—C30—H30C	109.5
N2—C11—N1	109.73 (19)	H30A—C30—H30C	109.5
N2—C11—C6	123.1 (2)	H30B—C30—H30C	109.5

C12—N3—N2—C11	0.7 (2)	C11—N1—C12—S1	−178.65 (17)
C19—N3—N2—C11	176.39 (19)	C13—N1—C12—S1	4.6 (3)
C6—C1—C2—C3	−60.5 (3)	C12—N1—C13—C18	88.6 (3)
C6—C1—C2—C7	60.3 (3)	C11—N1—C13—C18	−87.3 (3)
C7—C2—C3—C4	−60.2 (3)	C12—N1—C13—C14	−83.7 (3)
C1—C2—C3—C4	60.6 (3)	C11—N1—C13—C14	100.5 (3)
C2—C3—C4—C10	60.1 (3)	C18—C13—C14—C15	−0.7 (4)
C2—C3—C4—C5	−59.9 (3)	N1—C13—C14—C15	171.5 (2)
C10—C4—C5—C6	−60.3 (3)	C13—C14—C15—C16	−0.2 (4)
C3—C4—C5—C6	60.2 (3)	C14—C15—C16—C17	1.5 (4)
C4—C5—C6—C11	179.3 (2)	C14—C15—C16—F1	−176.4 (2)
C4—C5—C6—C1	−59.1 (3)	C15—C16—C17—C18	−2.0 (4)
C4—C5—C6—C9	58.5 (3)	F1—C16—C17—C18	176.0 (2)
C2—C1—C6—C11	178.4 (2)	C14—C13—C18—C17	0.3 (4)
C2—C1—C6—C5	58.6 (3)	N1—C13—C18—C17	−172.0 (2)
C2—C1—C6—C9	−58.8 (3)	C16—C17—C18—C13	1.0 (4)
C3—C2—C7—C8	60.3 (3)	C23—N4—C19—N3	75.5 (3)
C1—C2—C7—C8	−61.2 (3)	C20—N4—C19—N3	−52.4 (3)
C2—C7—C8—C10	−59.4 (3)	C12—N3—C19—N4	104.2 (3)
C2—C7—C8—C9	60.7 (3)	N2—N3—C19—N4	−71.0 (3)
C7—C8—C9—C6	−59.9 (3)	C19—N4—C20—C21	−172.94 (19)
C10—C8—C9—C6	60.6 (3)	C23—N4—C20—C21	57.0 (2)
C11—C6—C9—C8	−178.1 (2)	C24—N5—C21—C20	−172.6 (2)
C5—C6—C9—C8	−58.6 (3)	C22—N5—C21—C20	57.1 (3)
C1—C6—C9—C8	58.7 (3)	N4—C20—C21—N5	−56.0 (3)
C3—C4—C10—C8	−59.0 (3)	C24—N5—C22—C23	168.36 (19)
C5—C4—C10—C8	61.0 (3)	C21—N5—C22—C23	−59.8 (2)
C7—C8—C10—C4	58.6 (3)	C19—N4—C23—C22	171.58 (19)
C9—C8—C10—C4	−61.4 (3)	C20—N4—C23—C22	−58.7 (2)
N3—N2—C11—N1	0.3 (2)	N5—C22—C23—N4	60.5 (2)
N3—N2—C11—C6	−177.88 (18)	C21—N5—C24—C25	−13.9 (3)
C12—N1—C11—N2	−1.1 (2)	C22—N5—C24—C25	114.7 (3)
C13—N1—C11—N2	175.1 (2)	C21—N5—C24—C29	165.9 (2)
C12—N1—C11—C6	177.0 (2)	C22—N5—C24—C29	−65.5 (3)
C13—N1—C11—C6	−6.8 (4)	C29—C24—C25—C26	3.0 (4)
C5—C6—C11—N2	−3.9 (3)	N5—C24—C25—C26	−177.2 (2)
C1—C6—C11—N2	−123.3 (2)	C24—C25—C26—C27	0.6 (4)
C9—C6—C11—N2	115.2 (2)	C25—C26—C27—C28	−3.1 (4)

C5—C6—C11—N1	178.3 (2)	C26—C27—C28—C29	2.0 (4)
C1—C6—C11—N1	58.8 (3)	C30—O1—C29—C28	−2.9 (3)
C9—C6—C11—N1	−62.6 (3)	C30—O1—C29—C24	176.8 (2)
N2—N3—C12—N1	−1.3 (2)	C27—C28—C29—O1	−178.5 (2)
C19—N3—C12—N1	−176.79 (19)	C27—C28—C29—C24	1.7 (4)
N2—N3—C12—S1	178.75 (16)	C25—C24—C29—O1	176.1 (2)
C19—N3—C12—S1	3.3 (3)	N5—C24—C29—O1	−3.7 (3)
C11—N1—C12—N3	1.4 (2)	C25—C24—C29—C28	−4.2 (3)
C13—N1—C12—N3	−175.31 (18)	N5—C24—C29—C28	176.1 (2)

Hydrogen-bond geometry (Å, º)

Cg1 and Cg8 are the centroids of rings (N1–N3/C11/C12) and (C24–C29), respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C21—H21*A*···F1^{i}	0.97	2.47	3.407 (3)	162
C18—H18*A*···*Cg*1^{ii}	0.93	2.81	3.661	152
C9—H9*A*···*Cg*8^{iii}	0.97	2.80	3.697	155

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