Acta Cryst C
STRUCTURAL
CHEMISTRY

ISSN 2053-2296

Dihydrooxazolones and dihydroimidazolones derived from acylglycines: syntheses, molecular structures and supramolecular assembly

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Received 14 July 2015
Accepted 17 July 2015

Edited by A. L. Spek, Utrecht University, The Netherlands

Keywords: oxazolones; imidazolones; orientational disorder; crystal structure; hydrogen bonding; Erlenmeyer azlactones; supramolecular assembly.

CCDC references: 1413466; 1413465; 1413464; 1413463; 1040331; 1413462

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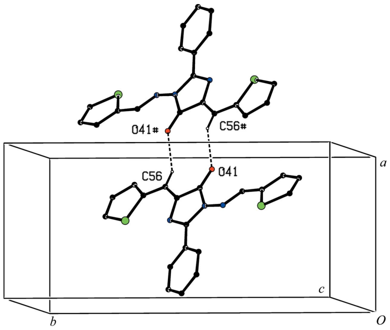
Syntheses and structures are described for some alkylidene-substituted dihydrooxazolones and dihydroimidazoles derived from simple acylglycines. A second, triclinic, polymorph of 4-benzylidene-2-(4-methylphenyl)-1,3-oxazol-5(4*H*)-one, $C_{17}H_{13}NO_2$, (I), has been identified and the structure of 2-methyl-4-[(thiophen-2-yl)methylidene]-1,3-oxazol-5(4*H*)-one, $C_9H_7NO_2S$, (II), has been rerefined taking into account the orientational disorder of the thienyl group in each of the two independent molecules. The reactions of phenylhydrazine with 2-phenyl-4-[(thiophen-2-yl)methylidene]-1,3-oxazol-5(4*H*)-one or 2-(4-methylphenyl)-4-[(thiophen-2-yl)methylidene]-1,3-oxazol-5(4*H*)-one yield, respectively, 3-anilino-2-phenyl-5-[(thiophen-2-yl)methylidene]-3,5-dihydro-4*H*-imidazol-4-one, $C_{10}H_{15}N_3OS$, (III), and 3-anilino-2-(4-methylphenyl)-5-[(thiophen-2-yl)methylidene]-3,5-dihydro-4*H*-imidazol-4-one, $C_{21}H_{17}N_3OS$, (IV), which both exhibit orientational disorder in their thienyl groups. The reactions of 2-phenyl-4-[(thiophen-2-yl)methylidene]-1,3-oxazol-5(4*H*)-one with hydrazine hydrate or with water yield, respectively, *N*-[3-hydrazinyl-3-oxo-1-(thiophen-2-yl)prop-1-en-2-yl]benzamide and 2-(benzoylamino)-3-(thiophen-2-yl)prop-2-enoic acid, which in turn react, respectively, with thiophene-2-carbaldehyde to form 2-phenyl-5-[(thiophen-2-yl)methylidene]-3-{[(*E*)-(thiophen-2-yl)methylidene]amino}-3,5-dihydro-4*H*-imidazol-4-one, $C_{19}H_{13}N_3OS_2$, (V), which exhibits orientational disorder in only one of its thienyl groups, and with methanol to give methyl (2*Z*)-2-(benzoylamino)-3-(thiophen-2-yl)prop-2-enoate, $C_{15}H_{13}NO_3S$, (VI). There are no direction-specific intermolecular interactions in the crystal structure of the triclinic polymorph of (I), but the molecules of (II) are linked by two independent C—H$\cdots$O hydrogen bonds to form $C_2^2(14)$ chains. Compounds (III) and (IV) both form centrosymmetric $R_2^2(10)$ dimers built from N—H$\cdots$O hydrogen bonds, while compound (V) forms a centrosymmetric $R_2^2(10)$ dimer built from C—H$\cdots$O hydrogen bonds. In the structure of compound (VI), a combination of N—H$\cdots$O and C—H$\cdots\pi$(arene) hydrogen bonds links the molecules into sheets. Comparisons are made with some similar compounds.



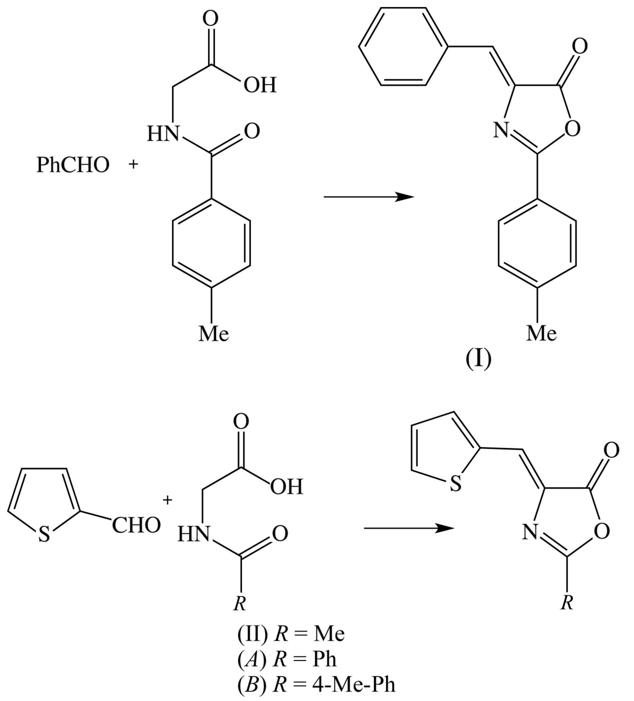
1. Introduction

Substituted 1,3-oxazol-5(4*H*)-ones, sometimes known as Erlenmeyer azlactones, are of value as intermediates in the synthesis of biologically active peptides, herbicides, fungicides and agrochemical intermediates (Reed & Kingston, 1986; El-Mekabaty, 2013), while unsaturated imidazolinones exhibit a wide range of pharmaceutical activities, including antimicrobial activity (Patel *et al.*, 2006; Suthakaran *et al.*, 2008), anthelmintic activity (Patel *et al.*, 2010), antibacterial activity (Hussein & Al-Tamamy, 2010) and anticonvulsant activity (Mohamed *et al.*, 2012). We report here the synthesis of a number of such compounds, all derived from simple acylglycine precursors, together with their molecular and supramolecular structures.

2. Experimental

2.1. Synthesis and crystallization

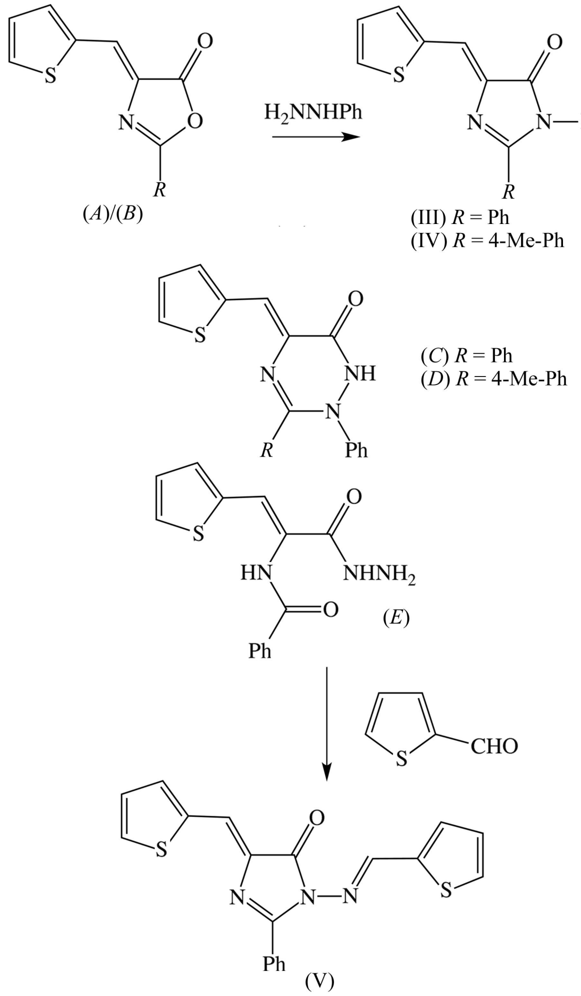
2.1.1. Preparation of compound (I). Compound (I) was prepared as described previously (Asiri *et al.*, 2012) but recrystallized from a mixture of acetone and toluene (1:1 *v*/*v*), rather than from ethanol as used previously, to give the triclinic polymorph instead of the original orthorhombic polymorph (see Scheme 1).



Scheme 1

2.1.2. Preparation of compounds (III) and (IV). For the syntheses of compounds (III) and (IV), mixtures of phenylhydrazine (0.01 mol) in glacial acetic acid (30 ml) with either 2-phenyl-4-[(thiophen-2-yl)methylidene]-1,3-oxazol-5(4*H*)-one [see (*A*) in Scheme 2] (0.01 mol) for (III) or 2-(4-methylphenyl)-4-[(thiophen-2-yl)methylidene]-1,3-oxazol-5(4*H*)-one [see (*B*) in Scheme 2] (0.01 mol) for (IV), themselves prepared exactly as for compound (II) (Sharma *et al.*, 2015), were heated under reflux for 3 h (see Scheme 2). The mixtures were allowed to cool to ambient temperature and were then poured into cold water. The resulting solid products (III) and (IV) were collected by filtration, washed with cold water and crystallized, at ambient temperature and in the presence of air, from mixtures of methanol and *N*,*N*-dimethylformamide (1:1 *v*/*v*) [m.p. 481–482 K for (III) and 459–460 K for (IV)].

2.1.3. Preparation of compound (V). For the synthesis of compound (V), a mixture of 2-phenyl-4-[(thiophen-2-yl)methylidene]-1,3-oxazol-5(4*H*)-one (0.01 mol) and hydrazine hydrate (0.022 mol) in dry ethanol (25 ml) was stirred at ambient temperature for 1 h. The resulting precipitate of *N*-[3-hydrazinyl-3-oxo-1-(thiophen-2-yl)prop-1-en-2-yl]benzamide was collected by filtration, washed with water and crystallized from ethanol (m.p. 467–468 K). A mixture of this hydrazine (0.01 mol) and thiophene-2-carbaldehyde (0.01 mol) in 2-propanol (30 ml) was heated under reflux for 8 h (see Scheme 2). The mixture was then cooled to ambient temperature and poured into cold water; the resulting solid product (V) was collected by filtration, washed with cold water and crystallized, at ambient temperature and in the presence of air, from a mixture of methanol and *N*,*N*-dimethylformamide (1:1 *v*/*v*) (m.p. 449 K).



Scheme 2

2.1.4. Preparation of compound (VI). For the synthesis of compound (VI) (see Scheme 3), 2-phenyl-4-[(thiophen-2-yl)methylidene]-1,3-oxazol-5(4*H*)-one (0.01 mol) was dissolved in a mixture of acetone and water (1:1 *v*/*v*, 50 ml), and this solution was heated under reflux for 8 h. The mixture was then cooled to ambient temperature and the resulting precipitate of 2-(benzoylamino)-3-(thiophen-2-yl)prop-2-enoic acid (*F*) was

Table 1
Experimental details.

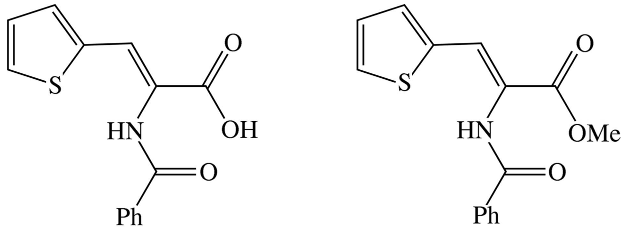
The diffractometer used for all determinations was a Bruker APEXII CCD diffractometer.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{17}H_{13}NO_2$	$C_9H_7NO_2S$	$C_{20}H_{15}N_3OS$
M_r	263.28	193.22	345.41
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/n$	Triclinic, $P\overline{1}$
Temperature (K)	296	293	296
a, b, c (Å)	7.9940 (4), 9.0648 (4), 10.7692 (6)	12.2264 (11), 9.8581 (7), 15.8735 (13)	8.8460 (2), 9.9951 (2), 11.2435 (2)
α, β, γ (°)	109.136 (4), 109.397 (4), 97.320 (4)	90, 112.129 (10), 90	114.481 (1), 98.564 (1), 105.891 (1)
V (Å^3)	670.52 (6)	1772.3 (3)	830.15 (3)
Z	2	8	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.09	0.33	0.21
Crystal size (mm)	0.20 × 0.15 × 0.15	0.30 × 0.20 × 0.20	0.35 × 0.20 × 0.15
Data collection			
Absorption correction	–	Multi-scan (*SADABS*; Sheldrick, 2003)	Multi-scan (*SADABS*; Sheldrick, 2003)
T_{min}, T_{max}	–	0.788, 0.937	0.917, 0.969
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10476, 2677, 1604	7625, 3916, 2656	22029, 4814, 3747
R_{int}	0.043	0.030	0.022
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.629	0.651	0.703
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.052, 0.150, 1.00	0.044, 0.111, 1.03	0.041, 0.113, 1.01
No. of reflections	2677	3916	4814
No. of parameters	182	269	245
No. of restraints	0	26	13
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.21	0.19, −0.21	0.28, −0.26

	(IV)	(V)	(VI)
Crystal data			
Chemical formula	$C_{21}H_{17}N_3OS$	$C_{19}H_{13}N_3OS_2$	$C_{15}H_{13}NO_3S$
M_r	359.44	363.44	287.32
Crystal system, space group	Triclinic, $P\overline{1}$	Orthorhombic, *Pbca*	Orthorhombic, *Pbca*
Temperature (K)	296	296	296
a, b, c (Å)	9.1697 (5), 10.7939 (5), 11.0837 (6)	8.2717 (2), 17.4530 (3), 23.3501 (5)	16.6123 (3), 9.7589 (2), 17.7543 (4)
α, β, γ (°)	66.424 (2), 66.147 (3), 67.934 (2)	90, 90, 90	90, 90, 90
V (Å^3)	889.13 (8)	3370.96 (12)	2878.29 (10)
Z	2	8	8
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.20	0.33	0.23
Crystal size (mm)	0.22 × 0.20 × 0.15	0.45 × 0.45 × 0.45	0.45 × 0.25 × 0.15
Data collection			
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2003)	Multi-scan (*SADABS*; Sheldrick, 2003)	Multi-scan (*SADABS*; Sheldrick, 2003)
T_{min}, T_{max}	0.911, 0.971	0.817, 0.863	0.881, 0.966
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	33541, 4395, 3909	24050, 4912, 3966	18087, 3292, 2439
R_{int}	0.023	0.021	0.023
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.669	0.703	0.650
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.100, 1.14	0.043, 0.120, 1.02	0.045, 0.118, 1.07
No. of reflections	4395	4912	3292
No. of parameters	255	242	185
No. of restraints	13	13	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.28	0.33, −0.33	0.27, −0.40

Computer programs: *APEX2* (Bruker, 2007), *SAINT* (Bruker, 2007), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

collected by filtration, dried and crystallized from ethanol (m.p. 465–466 K). A solution of this acid (0.01 mol) in methanol (30 ml) containing a catalytic quantity of glacial acetic acid was heated under reflux for 8 h. The mixture was then cooled to ambient temperature and poured into cold water; the resulting solid product (VI) was collected by filtration, washed with cold water and crystallized, at ambient temperature and in the presence of air, from a mixture of methanol and *N*,*N*-dimethylformamide (1:1 *v*/*v*) (m.p. 435–436 K).



(*F*) (VI)

Scheme 3

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. For both of the independent molecules of compound (II), the disorder of the thienyl groups was modelled using two sets of atomic sites having unequal occupancies, and which were, in each case, related to one another by an approximate 180° rotation about the exocyclic C—C bond. For the two minor-occupancy components, the bonded distances and the one-angle nonbonded distances were restrained to be the same as the corresponding distances in the major-occupancy components, subject to uncertainties of 0.005 Å and 0.01 Å, respectively. In addition, the anisotropic displacement parameters for pairs of partial-occupancy atoms occupying essentially the same physical space were constrained to be identical. Under these conditions, the occupancies for molecule 1 were 0.974 (2) and 0.026 (2), and those for molecule 2 were 0.931 (2) and 0.069 (2). The thienyl disorder in compounds (III)–(V) was handled in a similar manner, giving occupancies of 0.9005 (19) and 0.0995 (19) in (III), 0.866 (2) and 0.134 (2) in (IV), and 0.9763 (18) and 0.0257 (18) for the ring in (V) which is linked *via* atom C56. For compounds (I) and (VI), and for the major disorder components of compounds (II)–(V), the H atoms were located in difference maps; H atoms bonded to C atoms which had been located in this way were then treated as riding atoms in geometrically idealized positions, with C—H distances of 0.93 (alkenyl, aryl and heteroaryl) or 0.96 Å (methyl), and with $U_{\rm iso}({\rm H}) = kU_{\rm eq}({\rm C})$, where $k = 1.5$ for the methyl groups, which were permitted to rotate but not to tilt, and $k = 1.2$ for all other H atoms bonded to C atoms. The H atoms in the minor-disorder components were included in calculated positions, on the same basis as specified above. For H atoms bonded to N atoms, the atomic coordinates were refined with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm N})$ giving the N—H distances shown in Table 3.

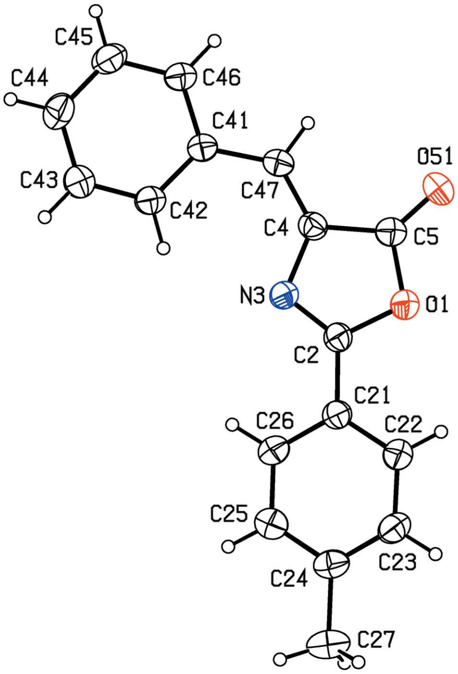


Figure 1
The molecular structure of compound (I) in the triclinic polymorph, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

3. Results and discussion

The reaction of 4-methylbenzoylglycine with benzaldehyde in the presence of acetic anhydride yields 4-benzylidene-2-(4-methylphenyl)-1,3-oxazol-5(4*H*)-one, (I) (Fig. 1), in a reaction which involves both the condensation of the aldehyde unit with the methylene group of the glycine and an intramolecular dehydration to form the oxazolone ring. This compound has been reported previously as an orthorhombic polymorph, in the space group *Pbca*, following crystallization from ethanol (Asiri *et al.*, 2012). We have now found a second polymorph, in the space group $P\overline{1}$, resulting from crystallization from a mixture of acetone and toluene. However, it is necessary to note here that in the original report (Asiri *et al.*, 2012), compound (I) was named, incorrectly, as the 2-phenyl, rather than the 2-(4-methylphenyl) derivative, and that the *Experimental* section of that report refers, rather surprisingly, to 4-methoxybenzoylglycine rather than to 4-methylbenzoylglycine.

An analogous reaction between acetylglycine and thiophene-2-carbaldehyde, also in the presence of acetic anhydride, gave 2-methyl-4-[(thiophen-2-yl)methylidene]-1,3-oxazol-5(4*H*)-one, (II), which was refined as a fully ordered structure, with $Z' = 2$ in the space group $P2_1/n$ (Sharma *et al.*, 2015). This fully ordered model led to some unexpected geometries, in particular, amongst the C—C distances of the thiophene rings, where, in one of the two independent rings, the lengths of the formal single C—C bond and of one of the formal double C═C bonds appeared to be identical within experimental uncertainty, suggesting the presence of unmodelled disorder of the usual thiophene type, involving disorder over two sets of atomic sites related by an approximate 180°

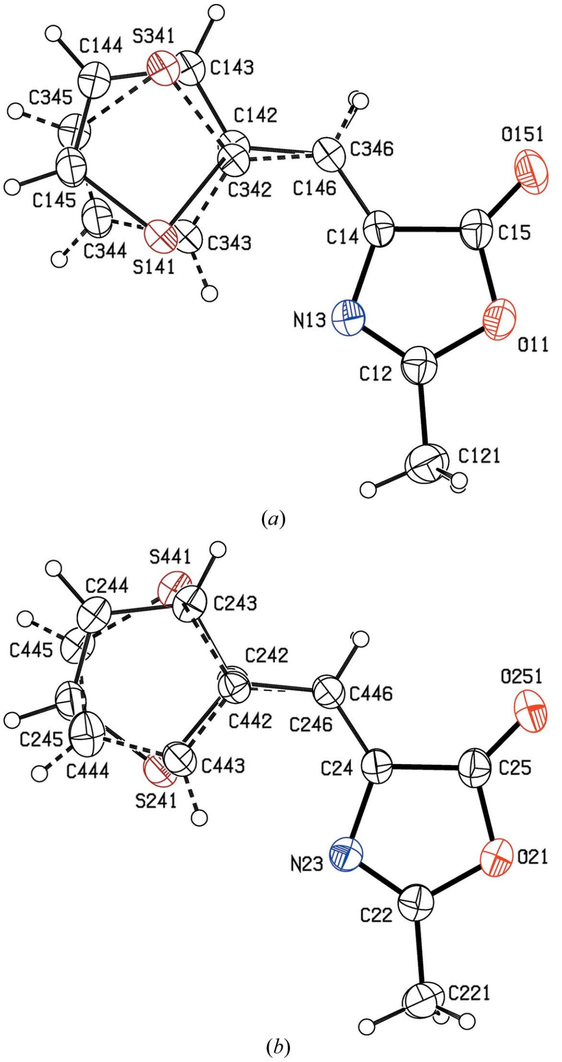


Figure 2

The molecular structures of the two independent and disordered molecules in compound (II), showing the atom-labelling schemes, for (*a*) a type 1 molecule where the site occupancies of the disorder components are 0.974 (2) and 0.026 (2), and (*b*) a type 2 molecule where the site occupancies of the disorder components are 0.931 (2) and 0.069 (2). The atomic coordinates of sites C146 and C346 are identical, as are those of sites C246 and C446, although those of the associated H atoms are not identical. Displacement ellipsoids are drawn at the 30% probability level.

rotation about the exocyclic C—C bond. Accordingly, we have now undertaken a new refinement of this structure using the original data set, but accounting for the previously unmodelled orientational disorder of the thiophene units in each of the two crystallographically independent molecules, where the site occupancies for the disorder components are 0.974 (2) and 0.026 (2) in molecule 1 (Fig. 2*a*), and 0.931 (2) and 0.069 (2) in molecule 2 (Fig. 2*b*). Similar reactions between thiophene-2-carbaldehyde and *N*-benzoylglycine and *N*-(4-methylbenzoyl)glycine yield oxazolones (*A*) and (*B*) (see Scheme 1), which are key intermediates in the synthesis of substituted alkylideneimidazolones.

Thus, in reactions with phenylhydrazine in anhydrous acetic acid, the intermediates (*A*) and (*B*) yield, respectively, 3-anilino-2-phenyl-5-[(thiophen-2-yl)methylidene]-3,5-dihydro-4*H*-imidazol-4-one, (III) (Fig. 3), and 3-anilino-2-(4-methylphenyl)-5-[(thiophen-2-yl)methylidene]-3,5-dihydro-4*H*-imidazol-4-one, (IV) (Fig. 4). In these two reactions, it is the terminal NH_2 group of phenylhydrazine rather than the less nucleophilic NH group which acts as the nucleophile towards the carbonyl group in the oxazolone ring, so forming imidazolones (III) and (IV) rather than 1,2,4-triazinones (*C*) and (*D*) (see Scheme 2). By contrast, the reaction of oxazolone (*A*) with hydrazine hydrate in ethanol, *i.e.* under conditions

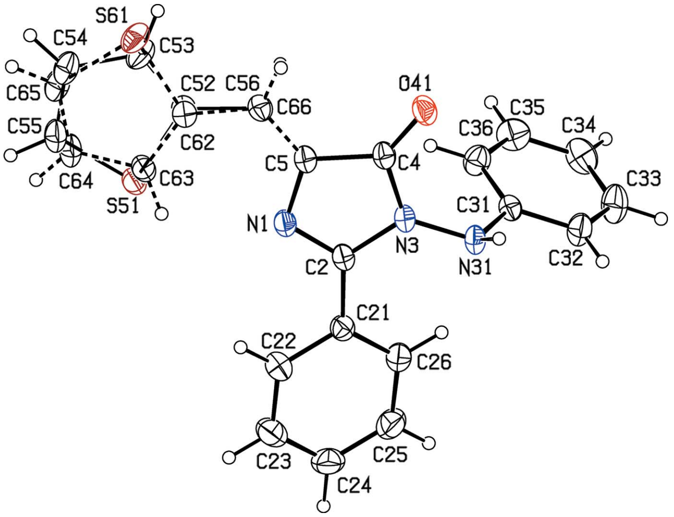


Figure 3

The molecular structure of compound (III), showing the atom-labelling scheme. The site occupancies of the disorder components are 0.9005 (19) and 0.0995 (19), and the atomic coordinates of sites C56 and C66 are identical, although those of the associated H atoms are not. Displacement ellipsoids are drawn at the 30% probability level.

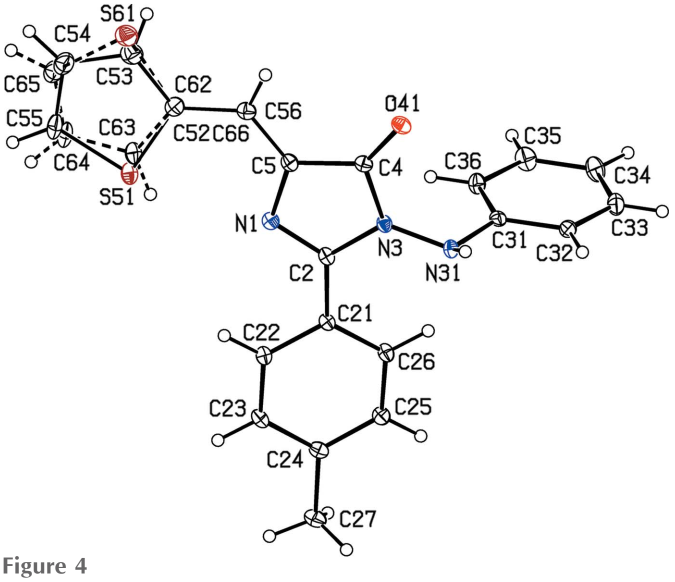


Figure 4

The molecular structure of compound (IV), showing the atom-labelling scheme. The site occupancies of the disorder components are 0.866 (2) and 0.134 (2), and the atomic coordinates of sites C56 and C66 are identical, although those of the associated H atoms are not. Displacement ellipsoids are drawn at the 30% probability level.

Table 2
Selected geometric parameters (Å, °) for compounds (I)–(V).

Data for the orthorhombic polymorph of compound (I) are taken from Asiri *et al.* (2012), but with the atom-numbering modified to match the systematic numbering used for the triclinic polymorph.

(*a*) Oxazolones

Parameter	(I) Triclinic nil	(I) Orthorhombic nil	(II) Mol. 1 *x* = 1	(II) Mol. 2 *x* = 2
O*x*1—C*x*2	1.391 (2)	1.3895 (18)	1.385 (2)	1.389 (2)
C*x*2—N*x*3	1.283 (2)	1.2915 (19)	1.273 (3)	1.273 (3)
N*x*3—C*x*4	1.403 (2)	1.4017 (19)	1.407 (2)	1.403 (3)
C*x*4—C*x*5	1.465 (3)	1.475 (2)	1.454 (3)	1.467 (3)
C*x*5—O*x*1	1.391 (2)	1.3913 (19)	1.388 (3)	1.388 (3)
C*x*5—O*x*51	1.202 (2)	1.2028 (18)	1.194 (2)	1.196 (2)
C*x*4—C*x*46			1.337 (3)	1.333 (3)
C*x*4—C*x*47	1.346 (3)	1.348 (2)		
C*x*46—C*x*42			1.426 (3)	1.437 (2)
C*x*47—C*x*41	1.451 (3)	1.456 (2)		
C*x*4—C*x*46—C*x*42			128.66 (19)	128.5 (2)
C*x*4—C*x*47—C*x*41	129.84 (18)	129.62 (14)		
Dihedral 1	11.38 (14)	7.98 (8)		
Dihedral 2	5.35 (13)	5.50 (8)	2.5 (2)	4.8 (3)
Dihedral 3	16.70 (13)	13.36 (7)		

(*b*) Imidazolones

Parameter	(III)	(IV)	(V)
N1—C2	1.2996 (16)	1.3075 (17)	1.3043 (17)
C2—N3	1.4056 (15)	1.4067 (16)	1.4004 (17)
C3—C4	1.3842 (18)	1.3952 (17)	1.4040 (18)
C4—C5	1.4750 (17)	1.4702 (18)	1.473 (2)
C5—N1	1.3946 (17)	1.3974 (17)	1.3836 (19)
C4—O41	1.2225 (15)	1.2225 (16)	1.2117 (18)
N3—N31	1.3895 (14)	1.3893 (15)	1.3872 (16)
N31—C31	1.4216 (16)	1.4144 (17)	
N31—C36			1.2872 (19)
C36—C32			1.438 (2)
C5—C56	1.3469 (8)	1.3595 (19)	1.347 (2)
C56—C52	1.4357 (8)	1.433 (2)	1.429 (2)
C5—C56—C52	127.09 (14)	125.57 (18)	127.39 (15)
N3—N31—C36			116.94 (13)
N31—C36—C32			120.26 (14)
C2—N3—N31—C31	100.78 (14)	105.69 (14)	
N3—N31—C31—C32	−177.77 (12)	163.55 (11)	
C2—N3—N31—C36			162.33 (14)
N3—N31—C36—C32			179.49 (13)
N31—C36—C32—S31			1.9 (2)
Dihedral 4	20.44 (8)	17.68 (7)	16.51 (8)
Dihedral 5	7.25 (14)	7.3 (5)	8.64 (9)
Dihedral 6	82.35 (8)	89.36 (8)	20.09 (8)

Notes: ‘Dihedral 1’ represents the dihedral angle between the oxazolone ring and the ring linked to it at atom C2. ‘Dihedral 2’ represents the dihedral angle between the oxazolone ring and the major component of the ring linked to it *via* the methine bridge at atom C*x*4. ‘Dihedral 3’ represents the dihedral angle between the two rings linked to the oxazolone ring at atoms C2 and C4. ‘Dihedral 4’ represents the dihedral angle between the imidazolone ring and the ring linked to it at atom C2. ‘Dihedral 5’ represents the dihedral angle between the imidazolone ring and the major component of the ring linked to it *via* the methine bridge at atom C5. ‘Dihedral 6’ represents the dihedral angle between the imidazolone ring and the major component of the ring linked to it *via* atom N31.

not conducive to intramolecular dehydration reactions, gives acyl hydrazine (*E*) (see Scheme 2), subsequent reaction of which with thiophene-2-carbaldehyde gives 2-phenyl-5-[(thiophen-2-yl)methylidene]-3-{[(*E*)-(thiophen-2-yl)methylidene]amino}-3,5-dihydro-4*H*-imidazol-4-one, (V) (Fig. 5). Finally, simple hydrolysis of oxazolone (*A*) yields methylideneamino acid (*F*) (see Scheme 3), esterification of which with methanol gives methyl (2*Z*)-2-(benzoylamino)-3-(thiophen-2-yl)prop-2-enoate, (VI) (Fig. 6).

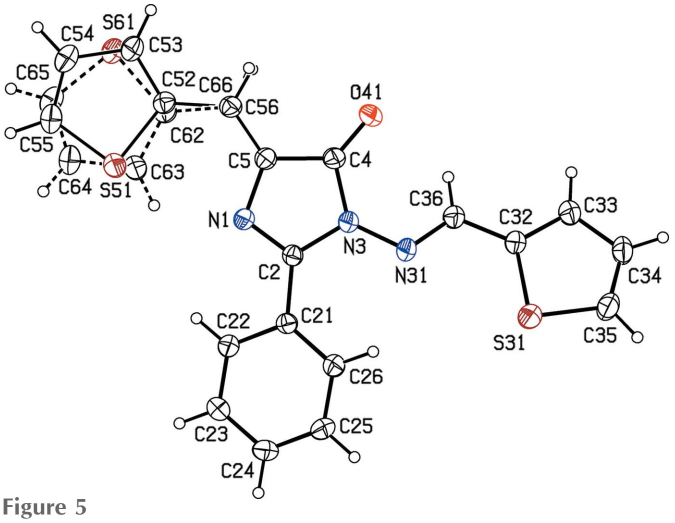


Figure 5
The molecular structure of compound (V), showing the atom-labelling scheme. The site occupancies of the disorder components for the thiophene ring bonded to atom C56 are 0.9763 (18) and 0.0237 (18). The atomic coordinates of sites C56 and C66 are identical, although those of the associated H atoms are not. Displacement ellipsoids are drawn at the 30% probability level.

As noted above, compound (I) crystallizes in two polymorphic forms, a triclinic form in the space group $P\overline{1}$ reported here and an orthorhombic form in the space group *Pbca* (Asiri

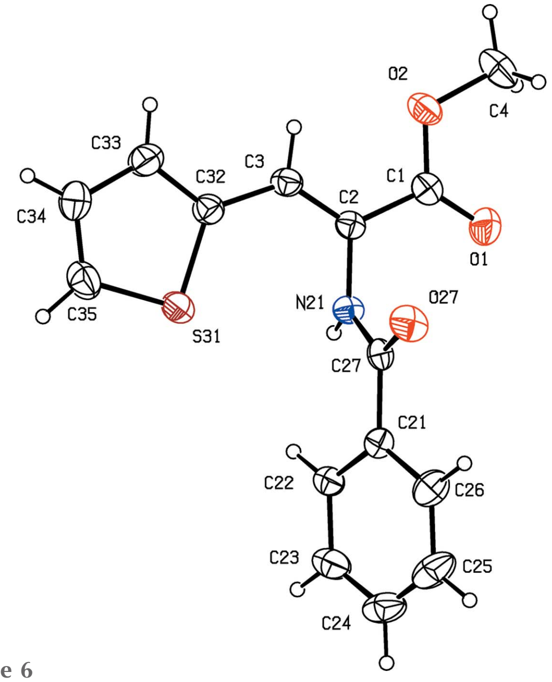


Figure 6
The molecular structure of compound (VI), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

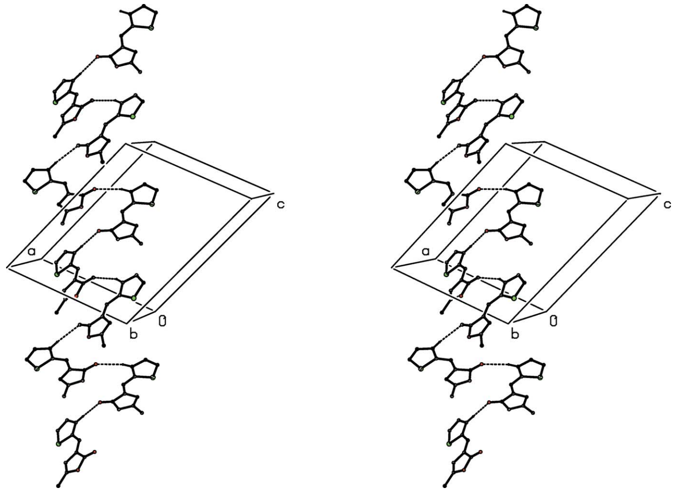


Figure 7
A stereoview of part of the crystal structure of compound (II), showing the formation of a hydrogen-bonded $C_2^2(14)$ chain running parallel to [101]. For the sake of clarity, the minor-disorder components and H atoms not involved in the motif shown have been omitted.

et al., 2012). There are no obvious similarities between any of the unit-cell dimensions for the two polymorphs and, as discussed below, the supramolecular assembly is different for the two forms. The constitutions of compounds (III) and (IV) are very similar, and they differ only in the presence of a methyl group in (IV) which is absent from (III). Although they both crystallize in the space group $P\overline{1}$ with fairly similar unit-cell repeat vectors, in compound (III), the inter-axial angles at the origin are all significantly greater than 90°, while those in compound (IV) are all significantly less than 90°. Hence, these two compounds cannot be even approximately isostructural. Their molecular structures, as opposed to their crystal structures are, however, quite similar, as discussed below; the reference molecules for compounds (III) and (IV) were selected to have the same configuration at amino atom N31 (Figs. 3 and 4).

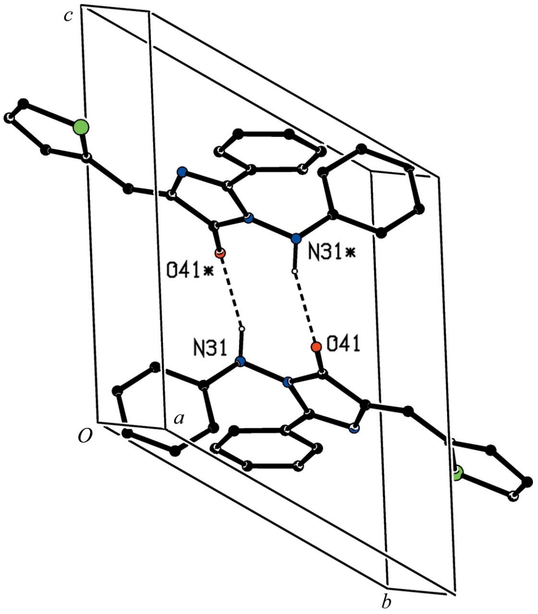


Figure 8
Part of the crystal structure of compound (III), showing the formation of a hydrogen-bonded $R_2^2(10)$ dimer centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. For the sake of clarity, the minor disorder components and H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(-x + 1, -y + 1, -z + 1)$. Note the different orientations of the unit-cell outlines in Figs. 8 and 9.

Compounds (II)–(V) exhibit orientational disorder of the thienyl units, in which the two disorder forms are related by a rotation of approximately 180° about the exocyclic C—C bonds (Figs. 2–5). In imidazolones (III) and (IV), the site occupancies of the two disorder forms are 0.9005 (19) and 0.0995 (19) in (III), and 0.866 (2) and 0.134 (2) in (IV); in (V), the occupancies for the disorder components of the thienyl ring linked to atom C56 (Fig. 5) are 0.9763 (18) and 0.0257 (18), although there was no disorder in the thienyl ring linked *via* atom C36, nor is there any disorder in compound (VI). While the occupancies of the minor components are sometimes quite small, if this disorder is ignored the apparent bond distances within such thienyl rings show some unsatisfactory values, particularly those of the formally single and double C—C bonds.

In the four independent oxazolone molecules discussed here, *i.e.* the two polymorphs of compound (I) and the two independent molecules in compound (II), the interatomic distances are very similar (Table 2) and indicate strong bond fixation in the oxazolone rings. The molecular conformations in the two polymorphs of (I) are also very similar, as are those of the independent molecules in (II). The three independent imidazolone rings in compounds (III)–(V) are also similar to one another, although the exocyclic C—O distances in (III)–(V) exceed those in (I) and (II), because of the amidic character in the imidazolone rings. The phenyl rings and the thienyl rings bonded *via* atom C56 in compounds (III)–(V) do not show major deviations from the planes of the imidazolone rings, but the rings in (III) and (IV) which are bonded *via* atom N31 are almost orthogonal to the imidazolone rings. On the other hand, in (V), the plane of the ring bonded *via* atom N31 makes a dihedral angle with that of the imidazolone ring of only 20.04 (9)°.

Despite the small dihedral angles between the planes of the oxazolone rings in compounds (I) and (II) and the rings bonded to them *via* the methine bridge, and between the imidazolone rings in compounds (III)–(V) and the rings linked *via* the methine bridge, in each of (I)–(V), the C—C—C angle at the methine bridge is significantly greater than the idealized value of 120°, and in several cases it approaches 130° (Table 2). Likewise, in compound (VI), the corresponding C2—C3—C32 angle (Fig. 6) is 129.15 (18)°. Similarly wide C—C—C angles at methine bridges between pairs of nearly coplanar rings were observed in an extensive series of arylmethylidene-substituted 2-thioxothiazolidin-4-ones (Delgado *et al.*, 2005, 2006; Insuasty *et al.*, 2012).

There are no direction-specific intermolecular interactions of any kind in the structure of the triclinic polymorph of compound (I). By contrast, in the orthorhombic polymorph (Asiri *et al.*, 2012), molecules related by the *a*-glide planes are

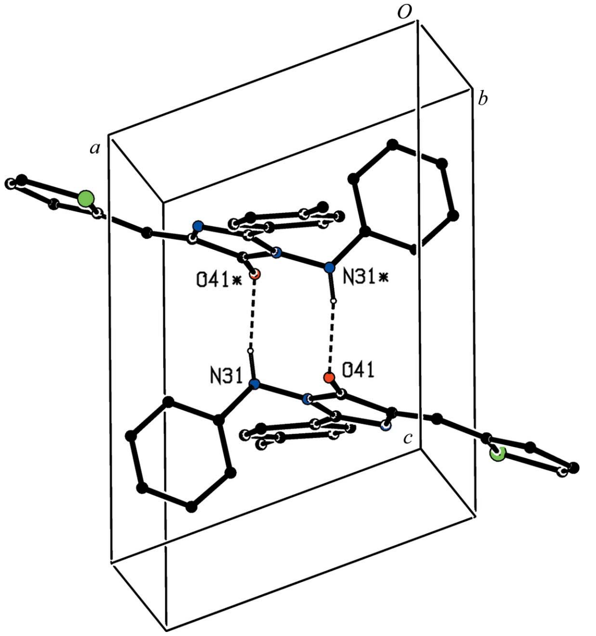


Figure 9
Part of the crystal structure of compound (IV), showing the formation of a hydrogen-bonded $R_2^2(10)$ dimer centred at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$). For the sake of clarity, the minor disorder components and H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) are at the symmetry position ($-x + 1$, $-y + 1$, $-z + 1$). Note the different orientations of the unit-cell outlines in Figs. 8 and 9.

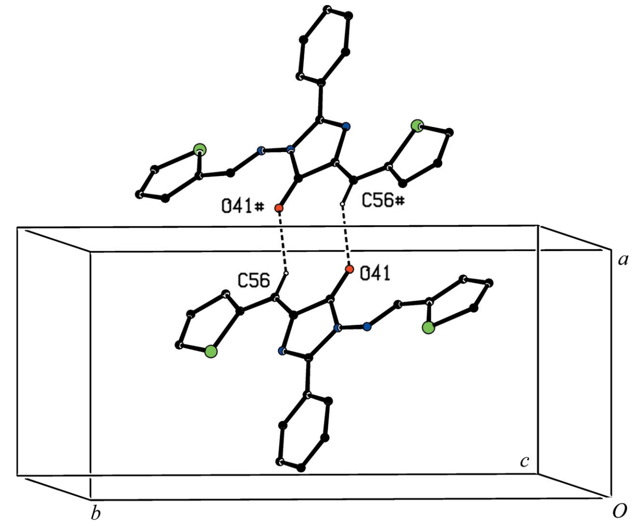


Figure 10
Part of the crystal structure of compound (V), showing the formation of a hydrogen-bonded $R_2^2(10)$ dimer centred at (1, $\frac{1}{2}$, $\frac{1}{2}$). For the sake of clarity, the minor disorder components and H atoms not involved in the motif shown have been omitted. Atoms marked with a hash (#) are at the symmetry position ($-x + 2$, $-y + 1$, $-z + 1$).

linked by C—H⋯O hydrogen bonds to form *C*(8) (Bernstein *et al.*, 1995) chains. The original report on this polymorph also refers to C—H⋯π(arene) hydrogen bonds and π–π stacking interactions which were said to contribute to the formation of a three-dimensional assembly. However, the C—H⋯π(arene) contacts mentioned both have H⋯(ring-centroid) distances greater than 2.90 Å and so are likely to be very weak, while the only short inter-ring contact involves the oxazolone ring which is by no means aromatic, so that this contact is likewise not structurally significant.

In the original report on compound (II), which used a fully ordered structural model (Sharma *et al.*, 2015), the two selected reference molecules did not form an asymmetric unit connected by a hydrogen bond; however, a simple rearrangement of the unit-cell contents provides an asymmetric unit in which the two selected molecules are linked by a C—H⋯O hydrogen bond (Table 3). Bimolecular aggregates of this type which are related by an *n*-glide plane are linked by a further C—H⋯O hydrogen bond to form a $C_2^2(14)$ chain running parallel to the [101] direction (Fig. 7). The only short C—H⋯π contact involves a methyl C—H bond and an H⋯(ring-centroid) distance of 2.95 Å, while the only short inter-ring contacts all involve oxazolone rings; none of these contacts is likely to be structurally significant.

Inversion-related pairs of molecules in each of compounds (III) and (IV) are linked by inversion-related pairs of almost linear N—H⋯O hydrogen bonds (Table 3) to form centrosymmetric dimers characterized in each case by an $R_2^2(10)$ motif centred at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$) (Figs. 8 and 9). However, these dimers are differently oriented within their respective unit cells, in part reflecting the different unit-cell angles for these two compounds. A cyclic centrosymmetric dimer of $R_2^2(10)$ type is also found in the structure of compound (V), but now built of C—H⋯O hydrogen bonds (Fig. 10), as opposed to the N—H⋯O hydrogen bonds in (III) and (IV). There are also C—H⋯π contacts (Table 3) in the structures of each of (III) and (V), but for both structures the H⋯(ring-centroid) distance is large and the C—H⋯(ring-centroid) angle is small (*cf.* Wood *et al.*, 2009), so that these contacts cannot be considered as structurally significant.

Table 3
Hydrogen bonds and short intermolecular contacts (Å, °) for compounds (II)–(VI).

*Cg*1 represents the centroid of the S51/C52–C55 ring and *Cg*2 represents the centroid of the C21–C26 ring.

Compound	*D*—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
(II)	C143—H143⋯O251	0.93	2.54	3.434 (4)	162
	C243—H243⋯O151^{i}	0.93	2.46	3.329 (9)	155
(III)	N31—H31⋯O41^{ii}	0.875 (17)	2.136 (18)	2.9767 (17)	161.1 (17)
	C34—H34⋯*Cg*1^{iii}	0.93	2.90	3.654 (2)	140
(IV)	N31—H31⋯O41^{ii}	0.876 (19)	2.010 (19)	2.8813 (16)	172.9 (19)
(V)	C56—H56⋯O41^{iv}	0.93	2.38	3.2270 (19)	151
	C35—H35⋯*Cg*2^{v}	0.93	2.99	3.7477 (19)	140
(VI)	N21—H21⋯O27^{vi}	0.83 (2)	2.01 (2)	2.8038 (19)	158.1 (19)
	C33—H33⋯*Cg*2^{vii}	0.93	2.68	3.472 (2)	144
	C35—H35⋯*Cg*2^{viii}	0.93	2.92	3.684 (2)	140

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{3}{2}, z + \frac{1}{2}$; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $-x + 1, -y + 1, -z$; (iv) $-x + 2, -y + 1, -z + 1$; (v) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$; (vi) $-x + \frac{1}{2}, -y + \frac{1}{2}, z$; (vii) $-x + \frac{1}{2}, -y + 1, z - \frac{1}{2}$; (viii) $-x, -y + 1, -z + 1$.

The supramolecular assembly in compound (VI) is more complex. Molecules related by the *b*-glide

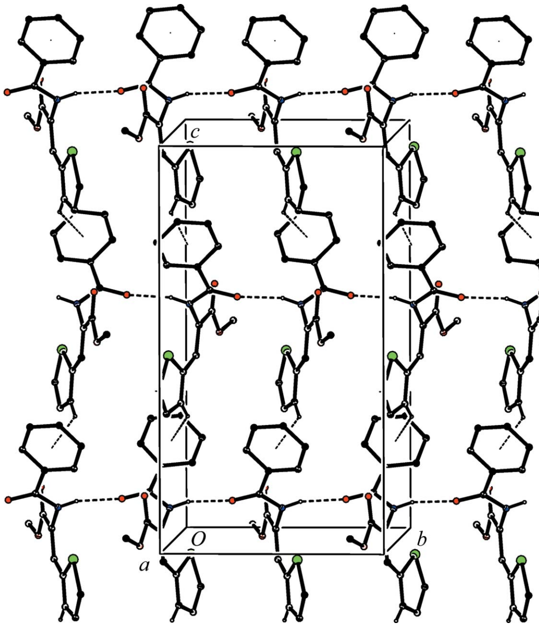


Figure 11
Part of the crystal structure of compound (VI), showing the formation of a sheet parallel to (100) formed by a combination of N—H⋯O and C—H⋯π(arene) hydrogen bonds. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

plane at $x = \frac{1}{4}$ are linked by N—H⋯O hydrogen bonds to form a *C*(4) chain running parallel to the [010] direction, and molecules related by the 2_1 screw axis along $(\frac{1}{4}, \frac{1}{2}, z)$ are linked by C—H⋯π(arene) hydrogen bonds to form a chain running parallel to the [001] direction. The combination of these two chain motifs generates a sheet lying parallel to (100) in the domain $0 < x < \frac{1}{2}$ (Fig. 11); a second sheet, related to the first by inversion, lies in the domain $\frac{1}{2} < x < 1$, but there are no direction-specific interactions between adjacent sheets.

Thus, the triclinic polymorph of compound (I) contains essentially isolated molecules, compounds (III)–(V) all form hydrogen-bonded dimers, the orthorhombic polymorph of (I) and compound (II) both form hydrogen-bonded chains, and compound (VI) forms hydrogen-bonded sheets, leading to a wide range of zero-, one- and two-dimensional supramolecular assembly types.

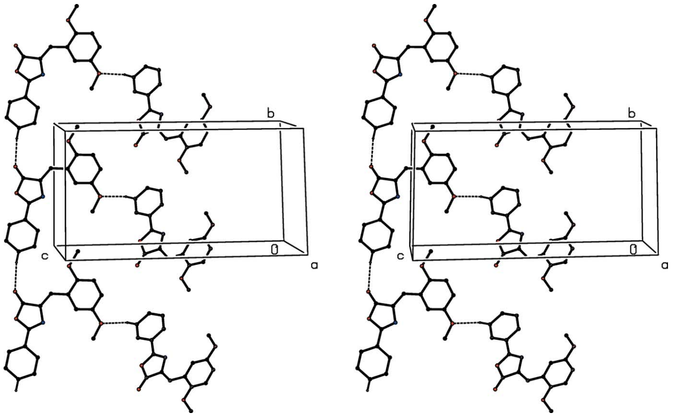
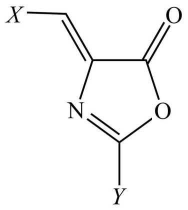
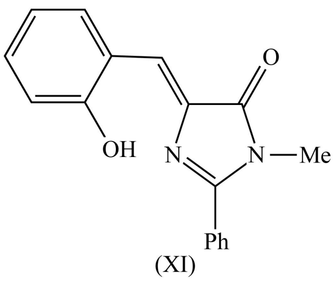


Figure 12
A stereoview of part of the crystal structure of compound (X), showing the formation of a hydrogen-bonded *C*(9) chain built from one type of molecule, from which molecules of the second type are pendent. The original atomic coordinates (Asiri & Ng, 2009) have been used and, for the sake of clarity, H atoms not involved in the motif shown have been omitted.

It is of interest briefly to note the supramolecular assembly of compounds (VII)–(XI) (see Scheme 4) in comparison with those of (I)–(VI) reported here. In each of compounds (VII) (Sun & Cui, 2008) and (VIII) (Gündoglu *et al.*, 2011*a*), pairs of molecules are linked by C—H⋯O hydrogen bonds to form centrosymmetric dimers. Compound (IX) (Gündoglu *et al.*, 2011*b*) crystallizes with $Z' = 2$ in the space group $P2_1/c$ and there is evidence from the C—C distances in the thienyl rings of unmodelled disordered in these rings. The two independent molecules differ in their supramolecular aggregation, although both form C—H⋯O hydrogen bonds; molecules of one type are linked into centrosymmetric dimers, as in (VII) and (VIII), while molecules of the second type are linked into chains. For compound (X), which crystallizes with $Z' = 2$ in the space group $P\overline{1}$, the supramolecular assembly is not mentioned in the original report (Asiri & Ng, 2009). In fact, molecules of one type, related by translation, are linked by C—H⋯O hydrogen bonds to form *C*(9) chains, from which the molecules of the other type are pendent (Fig. 12). Compound (XI) also crystallizes with $Z' = 2$ in the space group $P\overline{1}$ (Chang *et al.*, 2012); there is an intramolecular O—H⋯N hydrogen bond in each of the independent molecules, and molecules of both types form independent centrosymmetric dimers *via* C—H⋯O hydrogen bonds.



(VII) X = 3,4,5-trimethoxyphenyl, Y = phenyl
(VIII) $X = Y$ = 1-naphthyl
(IX) X = 2-thienyl, Y = 1-naphthyl
(X) X = 2,5-dimethoxyphenyl, Y = phenyl



(XI)

Scheme 4

Acknowledgements

The authors are indebted to the X-ray laboratory of Dicle University Scientific and Technological Applied and Research Center, Diyarbakir, Turkey, for use of the X-ray diffractometer. BN acknowledges the financial assistance of UGC through a BSR one-time grant for the purchase of chemicals. KNS gratefully acknowledges the Department of Chemistry, Shri Madhwa Vadiraja Institute of Technology, Bantakal (VTU, Belgaum), for providing access to research facilities.

References

Asiri, A. M., Faidallah, H. M., Sobahi, T. R., Ng, S. W. & Tiekink, E. R. T. (2012). *Acta Cryst.* E**68**, o1154.

Asiri, A. M. & Ng, S. W. (2009). *Acta Cryst.* E**65**, o1746.

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

Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chang, M.-J., Tsai, H.-Y., Fang, T.-C., Luo, M.-H. & Chen, K.-Y. (2012). *Acta Cryst.* E**68**, o902–o903.

Delgado, P., Quiroga, J., Cobo, J., Low, J. N. & Glidewell, C. (2005). *Acta Cryst.* C**61**, o477–o482.

Delgado, P., Quiroga, J., de la Torre, J. M., Cobo, J., Low, J. N. & Glidewell, C. (2006). *Acta Cryst.* C**62**, o382–o385.

El-Mekabaty, A. (2013). *Int. J. Modern Org. Chem.* **2**, 40–66.

Gündogdu, C., Alp, S., Ergün, Y., Tercan, B. & Hökelek, T. (2011*a*). *Acta Cryst.* E**67**, o1258–o1259.

Gündogdu, C., Alp, S., Ergün, Y., Tercan, B. & Hökelek, T. (2011*b*). *Acta Cryst.* E**67**, o1321–o1322.

Hussein, A. & Al-Tamamy, F. A. (2010). *Orient. J. Chem.* **26**, 421–427.

Insuasty, A., Insuasty, B., Cobo, J. & Glidewell, C. (2012). *Acta Cryst.* C**68**, o468–o471.

Mohamed, M. S., Mahmoud, R. K., Sayad, A. I. & El-Araby, M. E. (2012). *J. Med. Chem.* **2**, 24–29.

Patel, A., Bari, S., Talele, G., Patel, J. & Sarangapani, M. (2006). *Iran. J. Pharm. Res.* **4**, 249–254.

Patel, K., Jayachandran, E., Ravishah, V. & Sreenivasa, G. M. (2010). *Int. J. Pharm. Bio. Sci.* **1**, 1–13.

Reed, J. W. & Kingston, D. G. I. (1986). *J. Nat. Prod.* **49**, 626–630.

Sharma, P., Subbulakshmi, K. N., Narayana, B., Byrappa, K. & Kant, R. (2015). *Acta Cryst.* E**71**, o123–o124.

Sheldrick, G. M. (2003). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

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

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Sun, Y.-F. & Cui, Y.-P. (2008). *Acta Cryst.* E**64**, o678.

Suthakaran, R., Kavimani, S., Venkaiaiah, P. & Suganthi, K. (2008). *Rasayan J. Chem.* **1**, 22–29.

Wood, P. A., Allen, F. H. & Pidcock, E. (2009). *CrystEngComm*, **11**, 1563–1571.

supporting information

Acta Cryst. (2015). C**71**, 742-751 [doi:10.1107/S2053229615013637]

Dihydrooxazolones and dihydroimidazolones derived from acylglycines: syntheses, molecular structures and supramolecular assembly

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Computing details

For all compounds, data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

(I) 4-Benzylidene-2-(4-methylphenyl)-1,3-oxazol-5(4*H*)-one

Crystal data

$C_{17}H_{13}NO_2$
$M_r = 263.28$
Triclinic, $P\overline{1}$
$a = 7.9940$ (4) Å
$b = 9.0648$ (4) Å
$c = 10.7692$ (6) Å
$\alpha = 109.136$ (4)°
$\beta = 109.397$ (4)°
$\gamma = 97.320$ (4)°
$V = 670.52$ (6) Å^3
$Z = 2$
$F(000) = 276$
$D_x = 1.304$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3867 reflections
$\theta = 2.2$–30.6°
$\mu = 0.09$ mm^{-1}
$T = 296$ K
Block, yellow
0.20 × 0.15 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
10476 measured reflections
2677 independent reflections
1604 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.043$
$\theta_{\rm max} = 26.6°$, $\theta_{\rm min} = 2.2°$
$h = -10\rightarrow10$
$k = -11\rightarrow11$
$l = -13\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.150$
$S = 1.00$
2677 reflections
182 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0761P)^2 + 0.0683P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.16$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.70957 (17)	0.40830 (15)	0.36192 (16)	0.0593 (4)
C2	0.6065 (3)	0.5197 (2)	0.3523 (2)	0.0505 (5)
N3	0.4795 (2)	0.48433 (17)	0.22867 (19)	0.0521 (5)
C4	0.4878 (3)	0.3349 (2)	0.1400 (2)	0.0498 (5)
C5	0.6352 (3)	0.2843 (2)	0.2259 (2)	0.0571 (6)
O51	0.6940 (2)	0.16734 (17)	0.19946 (18)	0.0769 (5)
C21	0.6518 (3)	0.6603 (2)	0.4828 (2)	0.0506 (5)
C22	0.8097 (3)	0.6992 (2)	0.6047 (3)	0.0640 (6)
H22	0.8910	0.6344	0.6043	0.077*
C23	0.8463 (3)	0.8339 (3)	0.7263 (3)	0.0694 (7)
H23	0.9528	0.8585	0.8072	0.083*
C24	0.7297 (3)	0.9332 (2)	0.7316 (2)	0.0610 (6)
C25	0.5731 (3)	0.8932 (3)	0.6093 (3)	0.0686 (7)
H25	0.4915	0.9576	0.6100	0.082*
C26	0.5354 (3)	0.7603 (2)	0.4867 (2)	0.0631 (6)
H26	0.4301	0.7373	0.4054	0.076*
C27	0.7713 (3)	1.0800 (3)	0.8651 (3)	0.0835 (8)
H27A	0.9011	1.1308	0.9102	0.125*
H27B	0.7329	1.0478	0.9301	0.125*
H27C	0.7063	1.1551	0.8403	0.125*
C47	0.3884 (3)	0.2493 (2)	−0.0013 (2)	0.0525 (5)
H47	0.4138	0.1510	−0.0395	0.063*
C41	0.2476 (2)	0.2869 (2)	−0.1029 (2)	0.0497 (5)
C42	0.1943 (3)	0.4306 (2)	−0.0667 (2)	0.0588 (6)
H42	0.2483	0.5073	0.0272	0.071*
C43	0.0620 (3)	0.4589 (3)	−0.1697 (3)	0.0688 (7)
H43	0.0268	0.5547	−0.1446	0.083*
C44	−0.0188 (3)	0.3478 (3)	−0.3093 (3)	0.0705 (7)
H44	−0.1075	0.3686	−0.3782	0.085*
C45	0.0319 (3)	0.2058 (3)	−0.3466 (3)	0.0724 (7)
H45	−0.0231	0.1298	−0.4408	0.087*
C46	0.1641 (3)	0.1761 (2)	−0.2448 (2)	0.0616 (6)
H46	0.1983	0.0800	−0.2712	0.074*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0652 (8)	0.0530 (8)	0.0530 (10)	0.0193 (6)	0.0151 (7)	0.0203 (7)
C2	0.0557 (11)	0.0453 (10)	0.0510 (14)	0.0127 (8)	0.0189 (10)	0.0223 (10)

N3	0.0572 (9)	0.0476 (9)	0.0458 (12)	0.0134 (7)	0.0154 (9)	0.0169 (8)
C4	0.0553 (10)	0.0444 (10)	0.0512 (14)	0.0145 (8)	0.0225 (10)	0.0189 (10)
C5	0.0609 (12)	0.0518 (11)	0.0528 (15)	0.0142 (9)	0.0203 (11)	0.0163 (10)
O51	0.0874 (10)	0.0625 (9)	0.0763 (13)	0.0387 (8)	0.0253 (9)	0.0226 (9)
C21	0.0559 (11)	0.0461 (10)	0.0479 (14)	0.0108 (8)	0.0186 (10)	0.0197 (10)
C22	0.0701 (13)	0.0583 (12)	0.0551 (16)	0.0209 (10)	0.0151 (12)	0.0210 (11)
C23	0.0726 (13)	0.0659 (13)	0.0496 (16)	0.0127 (11)	0.0085 (11)	0.0167 (12)
C24	0.0724 (14)	0.0551 (11)	0.0496 (15)	0.0060 (10)	0.0262 (12)	0.0160 (10)
C25	0.0724 (14)	0.0641 (13)	0.0654 (17)	0.0230 (11)	0.0273 (13)	0.0192 (12)
C26	0.0596 (12)	0.0622 (12)	0.0503 (15)	0.0141 (10)	0.0116 (11)	0.0129 (11)
C27	0.0981 (18)	0.0723 (15)	0.0578 (18)	0.0059 (13)	0.0320 (15)	0.0044 (13)
C47	0.0583 (11)	0.0435 (10)	0.0524 (15)	0.0128 (8)	0.0234 (11)	0.0141 (10)
C41	0.0510 (10)	0.0472 (10)	0.0471 (14)	0.0094 (8)	0.0190 (9)	0.0159 (9)
C42	0.0680 (12)	0.0519 (11)	0.0491 (15)	0.0159 (10)	0.0180 (11)	0.0163 (10)
C43	0.0787 (14)	0.0605 (13)	0.0659 (18)	0.0239 (11)	0.0233 (13)	0.0269 (13)
C44	0.0684 (13)	0.0759 (15)	0.0623 (18)	0.0145 (11)	0.0135 (12)	0.0360 (13)
C45	0.0837 (15)	0.0652 (14)	0.0479 (15)	0.0101 (12)	0.0119 (12)	0.0161 (12)
C46	0.0682 (13)	0.0528 (11)	0.0501 (15)	0.0139 (10)	0.0164 (11)	0.0120 (11)

Geometric parameters (Å, º)

O1—C2	1.391 (2)	C26—H26	0.9300
O1—C5	1.391 (2)	C27—H27A	0.9600
C2—N3	1.283 (2)	C27—H27B	0.9600
C2—C21	1.449 (3)	C27—H27C	0.9600
N3—C4	1.403 (2)	C47—C41	1.451 (3)
C4—C47	1.346 (3)	C47—H47	0.9300
C4—C5	1.465 (3)	C41—C46	1.391 (3)
C5—O51	1.202 (2)	C41—C42	1.396 (3)
C21—C26	1.380 (3)	C42—C43	1.376 (3)
C21—C22	1.387 (3)	C42—H42	0.9300
C22—C23	1.378 (3)	C43—C44	1.374 (3)
C22—H22	0.9300	C43—H43	0.9300
C23—C24	1.377 (3)	C44—C45	1.373 (3)
C23—H23	0.9300	C44—H44	0.9300
C24—C25	1.382 (3)	C45—C46	1.374 (3)
C24—C27	1.505 (3)	C45—H45	0.9300
C25—C26	1.373 (3)	C46—H46	0.9300
C25—H25	0.9300		
C2—O1—C5	105.27 (15)	C21—C26—H26	119.6
N3—C2—O1	115.84 (17)	C24—C27—H27A	109.5
N3—C2—C21	127.46 (17)	C24—C27—H27B	109.5
O1—C2—C21	116.69 (18)	H27A—C27—H27B	109.5
C2—N3—C4	105.54 (16)	C24—C27—H27C	109.5
C47—C4—N3	128.79 (18)	H27A—C27—H27C	109.5
C47—C4—C5	122.91 (18)	H27B—C27—H27C	109.5
N3—C4—C5	108.24 (18)	C4—C47—C41	129.84 (18)

O51—C5—O1	121.5 (2)	C4—C47—H47	115.1
O51—C5—C4	133.4 (2)	C41—C47—H47	115.1
O1—C5—C4	105.09 (16)	C46—C41—C42	118.0 (2)
C26—C21—C22	118.38 (19)	C46—C41—C47	118.48 (18)
C26—C21—C2	119.35 (18)	C42—C41—C47	123.49 (19)
C22—C21—C2	122.26 (18)	C43—C42—C41	120.1 (2)
C23—C22—C21	120.1 (2)	C43—C42—H42	120.0
C23—C22—H22	120.0	C41—C42—H42	120.0
C21—C22—H22	120.0	C44—C43—C42	120.9 (2)
C24—C23—C22	121.9 (2)	C44—C43—H43	119.5
C24—C23—H23	119.1	C42—C43—H43	119.5
C22—C23—H23	119.1	C45—C44—C43	119.7 (2)
C23—C24—C25	117.4 (2)	C45—C44—H44	120.2
C23—C24—C27	121.4 (2)	C43—C44—H44	120.2
C25—C24—C27	121.2 (2)	C44—C45—C46	120.0 (2)
C26—C25—C24	121.4 (2)	C44—C45—H45	120.0
C26—C25—H25	119.3	C46—C45—H45	120.0
C24—C25—H25	119.3	C45—C46—C41	121.3 (2)
C25—C26—C21	120.8 (2)	C45—C46—H46	119.4
C25—C26—H26	119.6	C41—C46—H46	119.4

C5—O1—C2—N3	−1.0 (2)	C22—C23—C24—C25	−0.2 (4)
C5—O1—C2—C21	177.85 (16)	C22—C23—C24—C27	−180.0 (2)
O1—C2—N3—C4	0.5 (2)	C23—C24—C25—C26	−0.4 (3)
C21—C2—N3—C4	−178.25 (18)	C27—C24—C25—C26	179.4 (2)
C2—N3—C4—C47	−177.0 (2)	C24—C25—C26—C21	1.2 (4)
C2—N3—C4—C5	0.2 (2)	C22—C21—C26—C25	−1.3 (3)
C2—O1—C5—O51	−179.17 (19)	C2—C21—C26—C25	179.10 (19)
C2—O1—C5—C4	1.0 (2)	N3—C4—C47—C41	1.3 (4)
C47—C4—C5—O51	−3.2 (4)	C5—C4—C47—C41	−175.50 (18)
N3—C4—C5—O51	179.4 (2)	C4—C47—C41—C46	−179.7 (2)
C47—C4—C5—O1	176.57 (17)	C4—C47—C41—C42	1.9 (3)
N3—C4—C5—O1	−0.8 (2)	C46—C41—C42—C43	0.4 (3)
N3—C2—C21—C26	9.9 (3)	C47—C41—C42—C43	178.81 (19)
O1—C2—C21—C26	−168.84 (17)	C41—C42—C43—C44	−0.4 (3)
N3—C2—C21—C22	−169.7 (2)	C42—C43—C44—C45	0.5 (4)
O1—C2—C21—C22	11.6 (3)	C43—C44—C45—C46	−0.5 (4)
C26—C21—C22—C23	0.7 (3)	C44—C45—C46—C41	0.5 (4)
C2—C21—C22—C23	−179.7 (2)	C42—C41—C46—C45	−0.5 (3)
C21—C22—C23—C24	0.0 (4)	C47—C41—C46—C45	−179.0 (2)

(II) 2-Methyl-4-[(thiophen-2-yl)methylidene]-1,3-oxazol-5(4*H*)-one

Crystal data

$C_9H_7NO_2S$
$M_r = 193.22$
Monoclinic, $P2_1/n$
$a = 12.2264\ (11)$ Å
$b = 9.8581\ (7)$ Å
$c = 15.8735\ (13)$ Å
$\beta = 112.129\ (10)°$
$V = 1772.3\ (3)$ Å^3

$Z = 8$
$F(000) = 800$
$D_x = 1.448$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4062 reflections
$\theta = 3.6$–$29.0°$
$\mu = 0.33$ mm^{-1}
$T = 293$ K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.788$, $T_{\max} = 0.937$
7625 measured reflections
3916 independent reflections
2656 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.030$
$\theta_{\max} = 27.5°$, $\theta_{\min} = 3.9°$
$h = -15 \rightarrow 9$
$k = -12 \rightarrow 11$
$l = -20 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.111$
$S = 1.03$
3916 reflections
269 parameters
26 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0443P)^2 + 0.0864P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}

Special details

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

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

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
O11	0.45884 (13)	0.83638 (15)	0.00026 (10)	0.0577 (4)	
C12	0.5109 (2)	0.7836 (2)	−0.05649 (15)	0.0514 (6)	
N13	0.57969 (16)	0.68304 (18)	−0.02544 (12)	0.0489 (5)	
C14	0.57965 (18)	0.6603 (2)	0.06207 (14)	0.0439 (5)	
C15	0.50141 (19)	0.7585 (2)	0.07886 (16)	0.0511 (6)	
O151	0.47253 (14)	0.77891 (18)	0.14172 (11)	0.0687 (5)	
C121	0.4771 (2)	0.8499 (3)	−0.14588 (16)	0.0718 (7)	
H121	0.5148	0.8044	−0.1812	0.108*	
H122	0.5016	0.9431	−0.1376	0.108*	
H123	0.3930	0.8454	−0.1771	0.108*	
C146	0.64076 (19)	0.5661 (2)	0.12191 (14)	0.0455 (5)	0.974 (2)
H146	0.6310	0.5663	0.1772	0.055*	0.974 (2)
S141	0.74860 (5)	0.44227 (7)	0.01480 (4)	0.0492 (2)	0.974 (2)
C142	0.7189 (2)	0.4654 (2)	0.11224 (14)	0.0415 (5)	0.974 (2)
C143	0.7791 (4)	0.3751 (4)	0.1774 (2)	0.0561 (8)	0.974 (2)
H143	0.7744	0.3714	0.2344	0.067*	0.974 (2)

C144	0.8505 (2)	0.2865 (3)	0.14902 (17)	0.0589 (7)	0.974 (2)
H144	0.8979	0.2187	0.1855	0.071*	0.974 (2)
C145	0.8414 (2)	0.3120 (2)	0.06345 (16)	0.0548 (7)	0.974 (2)
H145	0.8818	0.2633	0.0341	0.066*	0.974 (2)
C346	0.64076 (19)	0.5661 (2)	0.12191 (14)	0.0455 (5)	0.026 (2)
H346	0.6342	0.5606	0.1783	0.055*	0.026 (2)
S341	0.803 (4)	0.353 (4)	0.174 (2)	0.0561 (8)	0.026 (2)
C342	0.716 (5)	0.474 (5)	0.0998 (19)	0.0415 (5)	0.026 (2)
C343	0.712 (7)	0.452 (6)	0.014 (2)	0.0492 (2)	0.026 (2)
H343	0.6828	0.5150	−0.0327	0.059*	0.026 (2)
C344	0.757 (6)	0.323 (5)	0.005 (3)	0.0548 (7)	0.026 (2)
H344	0.7449	0.2811	−0.0502	0.066*	0.026 (2)
C345	0.820 (7)	0.268 (5)	0.087 (3)	0.0589 (7)	0.026 (2)
H345	0.8681	0.1916	0.0944	0.071*	0.026 (2)
O21	0.61441 (14)	0.27829 (16)	0.36265 (9)	0.0560 (4)	
C22	0.5544 (2)	0.2366 (2)	0.41703 (15)	0.0498 (5)	
N23	0.57899 (16)	0.29992 (18)	0.49180 (11)	0.0465 (4)	
C24	0.66416 (19)	0.3967 (2)	0.49388 (13)	0.0435 (5)	
C25	0.6873 (2)	0.3832 (2)	0.41010 (15)	0.0526 (6)	
O251	0.75112 (16)	0.44151 (18)	0.38069 (11)	0.0743 (6)	
C221	0.4696 (2)	0.1256 (2)	0.37882 (16)	0.0659 (7)	
H211	0.4011	0.1601	0.3304	0.099*	
H212	0.4466	0.0882	0.4255	0.099*	
H213	0.5057	0.0562	0.3556	0.099*	
C246	0.71702 (18)	0.4874 (2)	0.55878 (13)	0.0441 (5)	0.931 (2)
H246	0.7734	0.5419	0.5492	0.053*	0.931 (2)
S241	0.59525 (6)	0.42819 (7)	0.67194 (4)	0.0498 (3)	0.931 (2)
C242	0.6984 (2)	0.5121 (2)	0.64157 (14)	0.0391 (6)	0.931 (2)
C243	0.7622 (7)	0.6011 (7)	0.7068 (4)	0.0554 (8)	0.931 (2)
H243	0.8227	0.6546	0.7029	0.066*	0.931 (2)
C244	0.7251 (2)	0.6029 (3)	0.78312 (18)	0.0540 (7)	0.931 (2)
H244	0.7588	0.6577	0.8341	0.065*	0.931 (2)
C245	0.6353 (3)	0.5151 (3)	0.77136 (17)	0.0532 (7)	0.931 (2)
H245	0.5995	0.5037	0.8132	0.064*	0.931 (2)
C446	0.71702 (18)	0.4874 (2)	0.55878 (13)	0.0441 (5)	0.069 (2)
H446	0.7604	0.5555	0.5451	0.053*	0.069 (2)
S441	0.769 (3)	0.621 (3)	0.7230 (14)	0.0554 (8)	0.069 (2)
C442	0.713 (3)	0.489 (3)	0.6480 (11)	0.0391 (6)	0.069 (2)
C443	0.639 (3)	0.411 (3)	0.6731 (17)	0.0498 (3)	0.069 (2)
H443	0.6009	0.3339	0.6414	0.060*	0.069 (2)
C444	0.627 (4)	0.460 (3)	0.755 (2)	0.0589 (7)	0.069 (2)
H444	0.5871	0.4143	0.7859	0.071*	0.069 (2)
C445	0.681 (4)	0.582 (4)	0.781 (2)	0.0540 (7)	0.069 (2)
H445	0.6711	0.6368	0.8247	0.065*	0.069 (2)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O11	0.0541 (10)	0.0528 (10)	0.0627 (10)	0.0129 (8)	0.0181 (8)	−0.0033 (9)
C12	0.0494 (13)	0.0499 (14)	0.0528 (13)	−0.0019 (12)	0.0169 (11)	−0.0059 (12)
N13	0.0529 (11)	0.0462 (11)	0.0487 (10)	0.0041 (10)	0.0205 (9)	−0.0022 (9)
C14	0.0414 (12)	0.0439 (13)	0.0469 (12)	0.0011 (11)	0.0171 (10)	−0.0064 (11)
C15	0.0428 (13)	0.0519 (14)	0.0574 (14)	0.0023 (11)	0.0174 (11)	−0.0089 (12)
O151	0.0661 (11)	0.0806 (12)	0.0675 (11)	0.0155 (10)	0.0343 (9)	−0.0121 (10)
C121	0.0816 (19)	0.0642 (17)	0.0649 (16)	0.0055 (15)	0.0223 (14)	0.0077 (14)
C146	0.0484 (13)	0.0481 (13)	0.0435 (12)	0.0005 (11)	0.0215 (10)	−0.0041 (11)
S141	0.0575 (4)	0.0540 (4)	0.0416 (3)	0.0067 (3)	0.0247 (3)	−0.0014 (3)
C142	0.0430 (11)	0.0434 (12)	0.0403 (11)	0.0003 (10)	0.0180 (10)	−0.0018 (10)
C143	0.063 (2)	0.063 (2)	0.0502 (14)	0.0172 (14)	0.0303 (13)	0.0056 (14)
C144	0.0673 (17)	0.0565 (15)	0.0599 (16)	0.0202 (14)	0.0318 (14)	0.0094 (13)
C145	0.0613 (16)	0.0501 (14)	0.0627 (16)	0.0103 (13)	0.0345 (13)	−0.0021 (13)
C346	0.0484 (13)	0.0481 (13)	0.0435 (12)	0.0005 (11)	0.0215 (10)	−0.0041 (11)
S341	0.063 (2)	0.063 (2)	0.0502 (14)	0.0172 (14)	0.0303 (13)	0.0056 (14)
C342	0.0430 (11)	0.0434 (12)	0.0403 (11)	0.0003 (10)	0.0180 (10)	−0.0018 (10)
C343	0.0575 (4)	0.0540 (4)	0.0416 (3)	0.0067 (3)	0.0247 (3)	−0.0014 (3)
C344	0.0613 (16)	0.0501 (14)	0.0627 (16)	0.0103 (13)	0.0345 (13)	−0.0021 (13)
C345	0.0673 (17)	0.0565 (15)	0.0599 (16)	0.0202 (14)	0.0318 (14)	0.0094 (13)
O21	0.0663 (10)	0.0586 (10)	0.0477 (9)	−0.0045 (9)	0.0268 (8)	−0.0098 (8)
C22	0.0524 (13)	0.0474 (13)	0.0497 (13)	0.0041 (11)	0.0192 (11)	0.0014 (11)
N23	0.0517 (11)	0.0453 (11)	0.0434 (10)	−0.0021 (9)	0.0189 (8)	−0.0014 (9)
C24	0.0466 (12)	0.0454 (12)	0.0420 (11)	0.0016 (11)	0.0206 (10)	0.0011 (11)
C25	0.0605 (15)	0.0536 (14)	0.0495 (13)	0.0044 (13)	0.0274 (12)	−0.0005 (12)
O251	0.0937 (14)	0.0812 (13)	0.0709 (11)	−0.0175 (11)	0.0572 (11)	−0.0084 (10)
C221	0.0703 (17)	0.0592 (16)	0.0636 (15)	−0.0111 (14)	0.0198 (13)	−0.0126 (14)
C246	0.0460 (12)	0.0437 (12)	0.0471 (12)	−0.0014 (11)	0.0226 (10)	0.0021 (11)
S241	0.0488 (4)	0.0555 (4)	0.0489 (4)	−0.0078 (3)	0.0228 (3)	0.0028 (3)
C242	0.0416 (13)	0.0350 (13)	0.0419 (11)	0.0027 (11)	0.0171 (10)	0.0044 (10)
C243	0.0629 (17)	0.056 (3)	0.051 (2)	−0.0061 (17)	0.026 (2)	−0.0113 (14)
C244	0.060 (2)	0.0577 (16)	0.0439 (13)	−0.0067 (14)	0.0190 (14)	−0.0093 (12)
C245	0.0629 (17)	0.0590 (18)	0.0456 (14)	−0.0004 (16)	0.0293 (13)	0.0018 (14)
C446	0.0460 (12)	0.0437 (12)	0.0471 (12)	−0.0014 (11)	0.0226 (10)	0.0021 (11)
S441	0.0629 (17)	0.056 (3)	0.051 (2)	−0.0061 (17)	0.026 (2)	−0.0113 (14)
C442	0.0416 (13)	0.0350 (13)	0.0419 (11)	0.0027 (11)	0.0171 (10)	0.0044 (10)
C443	0.0488 (4)	0.0555 (4)	0.0489 (4)	−0.0078 (3)	0.0228 (3)	0.0028 (3)
C444	0.0673 (17)	0.0565 (15)	0.0599 (16)	0.0202 (14)	0.0318 (14)	0.0094 (13)
C445	0.060 (2)	0.0577 (16)	0.0439 (13)	−0.0067 (14)	0.0190 (14)	−0.0093 (12)

Geometric parameters (Å, º)

O11—C12	1.385 (2)	O21—C25	1.388 (3)
O11—C15	1.388 (3)	O21—C22	1.389 (2)
C12—N13	1.273 (3)	C22—N23	1.273 (3)
C12—C121	1.473 (3)	C22—C221	1.472 (3)

N13—C14	1.407 (2)	N23—C24	1.403 (3)
C14—C146	1.337 (3)	C24—C246	1.333 (3)
C14—C15	1.454 (3)	C24—C25	1.467 (3)
C15—O151	1.194 (2)	C25—O251	1.196 (2)
C121—H121	0.9600	C221—H211	0.9600
C121—H122	0.9600	C221—H212	0.9600
C121—H123	0.9600	C221—H213	0.9600
C146—C142	1.426 (3)	C246—C242	1.437 (2)
C146—H146	0.9300	C246—H246	0.9300
S141—C145	1.695 (2)	S241—C245	1.698 (3)
S141—C142	1.7315 (19)	S241—C242	1.722 (2)
C142—C143	1.353 (4)	C242—C243	1.357 (5)
C143—C144	1.422 (4)	C243—C244	1.444 (5)
C143—H143	0.9300	C243—H243	0.9300
C144—C145	1.344 (3)	C244—C245	1.354 (3)
C144—H144	0.9300	C244—H244	0.9300
C145—H145	0.9300	C245—H245	0.9300
S341—C345	1.695 (5)	S441—C445	1.697 (6)
S341—C342	1.733 (5)	S441—C442	1.723 (5)
C342—C343	1.355 (6)	C442—C443	1.358 (7)
C343—C344	1.424 (6)	C443—C444	1.446 (7)
C343—H343	0.9300	C443—H443	0.9300
C344—C345	1.345 (6)	C444—C445	1.354 (6)
C344—H344	0.9300	C444—H444	0.9300
C345—H345	0.9300	C445—H445	0.9300
C12—O11—C15	105.55 (16)	C25—O21—C22	105.35 (16)
N13—C12—O11	116.05 (19)	N23—C22—O21	116.1 (2)
N13—C12—C121	128.5 (2)	N23—C22—C221	129.1 (2)
O11—C12—C121	115.4 (2)	O21—C22—C221	114.76 (19)
C12—N13—C14	105.09 (17)	C22—N23—C24	105.52 (17)
C146—C14—N13	127.16 (18)	C246—C24—N23	127.86 (17)
C146—C14—C15	124.20 (19)	C246—C24—C25	124.0 (2)
N13—C14—C15	108.64 (19)	N23—C24—C25	108.18 (18)
O151—C15—O11	121.9 (2)	O251—C25—O21	121.8 (2)
O151—C15—C14	133.4 (2)	O251—C25—C24	133.4 (2)
O11—C15—C14	104.67 (18)	O21—C25—C24	104.82 (18)
C12—C121—H121	109.5	C22—C221—H211	109.5
C12—C121—H122	109.5	C22—C221—H212	109.5
H121—C121—H122	109.5	H211—C221—H212	109.5
C12—C121—H123	109.5	C22—C221—H213	109.5
H121—C121—H123	109.5	H211—C221—H213	109.5
H122—C121—H123	109.5	H212—C221—H213	109.5
C14—C146—C142	128.66 (19)	C24—C246—C242	128.5 (2)
C14—C146—H146	115.7	C24—C246—H246	115.7
C142—C146—H146	115.7	C242—C246—H246	115.7
C145—S141—C142	91.29 (10)	C245—S241—C242	91.85 (12)
C143—C142—C146	124.7 (2)	C243—C242—C246	124.4 (3)

C143—C142—S141	111.06 (18)	C243—C242—S241	111.6 (3)
C146—C142—S141	124.23 (17)	C246—C242—S241	124.04 (18)
C142—C143—C144	112.6 (3)	C242—C243—C244	112.1 (4)
C142—C143—H143	123.7	C242—C243—H243	123.9
C144—C143—H143	123.7	C244—C243—H243	123.9
C145—C144—C143	112.2 (2)	C245—C244—C243	111.7 (3)
C145—C144—H144	123.9	C245—C244—H244	124.1
C143—C144—H144	123.9	C243—C244—H244	124.1
C144—C145—S141	112.84 (17)	C244—C245—S241	112.69 (18)
C144—C145—H145	123.6	C244—C245—H245	123.7
S141—C145—H145	123.6	S241—C245—H245	123.7
C345—S341—C342	91.3 (4)	C445—S441—C442	91.8 (4)
C343—C342—S341	110.5 (5)	C443—C442—S441	111.4 (5)
C342—C343—C344	112.0 (8)	C442—C443—C444	111.9 (6)
C342—C343—H343	124.0	C442—C443—H443	124.0
C344—C343—H343	124.0	C444—C443—H443	124.0
C345—C344—C343	111.6 (8)	C445—C444—C443	111.5 (7)
C345—C344—H344	124.2	C445—C444—H444	124.2
C343—C344—H344	124.2	C443—C444—H444	124.2
C344—C345—S341	112.5 (6)	C444—C445—S441	112.1 (6)
C344—C345—H345	123.8	C444—C445—H445	123.9
S341—C345—H345	123.8	S441—C445—H445	123.9

C15—O11—C12—N13	0.4 (3)	C25—O21—C22—N23	0.0 (2)
C15—O11—C12—C121	179.20 (19)	C25—O21—C22—C221	179.19 (19)
O11—C12—N13—C14	−0.7 (3)	O21—C22—N23—C24	−0.1 (2)
C121—C12—N13—C14	−179.3 (2)	C221—C22—N23—C24	−179.1 (2)
C12—N13—C14—C146	−178.8 (2)	C22—N23—C24—C246	179.9 (2)
C12—N13—C14—C15	0.7 (2)	C22—N23—C24—C25	0.1 (2)
C12—O11—C15—O151	−179.9 (2)	C22—O21—C25—O251	−179.8 (2)
C12—O11—C15—C14	0.0 (2)	C22—O21—C25—C24	0.1 (2)
C146—C14—C15—O151	−0.9 (4)	C246—C24—C25—O251	0.0 (4)
N13—C14—C15—O151	179.6 (2)	N23—C24—C25—O251	179.8 (3)
C146—C14—C15—O11	179.08 (19)	C246—C24—C25—O21	−179.9 (2)
N13—C14—C15—O11	−0.4 (2)	N23—C24—C25—O21	−0.1 (2)
N13—C14—C146—C142	−0.9 (4)	N23—C24—C246—C242	−1.9 (4)
C15—C14—C146—C142	179.7 (2)	C25—C24—C246—C242	177.9 (2)
C14—C146—C142—C143	178.7 (3)	C24—C246—C242—C243	175.4 (5)
C14—C146—C142—S141	−1.6 (4)	C24—C246—C242—S241	−2.4 (4)
C145—S141—C142—C143	−0.1 (3)	C245—S241—C242—C243	1.3 (4)
C145—S141—C142—C146	−179.8 (2)	C245—S241—C242—C246	179.3 (2)
C146—C142—C143—C144	180.0 (3)	C246—C242—C243—C244	−179.0 (4)
S141—C142—C143—C144	0.3 (4)	S241—C242—C243—C244	−1.0 (7)
C142—C143—C144—C145	−0.4 (5)	C242—C243—C244—C245	0.0 (7)
C143—C144—C145—S141	0.3 (4)	C243—C244—C245—S241	0.9 (5)
C142—S141—C145—C144	−0.1 (2)	C242—S241—C245—C244	−1.3 (2)
C345—S341—C342—C343	−5 (5)	C445—S441—C442—C443	−6 (3)
S341—C342—C343—C344	13 (5)	S441—C442—C443—C444	1 (4)

C342—C343—C344—C345	−16 (6)	C442—C443—C444—C445	7 (5)
C343—C344—C345—S341	12 (7)	C443—C444—C445—S441	−12 (5)
C342—S341—C345—C344	−4 (6)	C442—S441—C445—C444	10 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C143—H143···O251	0.93	2.54	3.434 (4)	162
C243—H243···O151^{i}	0.93	2.46	3.329 (9)	155

Symmetry code: (i) $x+1/2, -y+3/2, z+1/2$.

(III) 3-Anilino-2-phenyl-5-[(thiophen-2-yl)methylidene]-3,5-dihydro-4*H*-imidazol-4-one

Crystal data

$C_{20}H_{15}N_3OS$	$Z = 2$
$M_r = 345.41$	$F(000) = 360$
Triclinic, $P\overline{1}$	$D_x = 1.382$ Mg m^{-3}
$a = 8.8460 (2)$ Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 9.9951 (2)$ Å	Cell parameters from 7165 reflections
$c = 11.2435 (2)$ Å	$\theta = 2.1–35.0°$
$\alpha = 114.481 (1)°$	$\mu = 0.21$ mm^{-1}
$\beta = 98.564 (1)°$	$T = 296$ K
$\gamma = 105.891 (1)°$	Block, brown
$V = 830.15 (3)$ Å^3	0.35 × 0.20 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer	22029 measured reflections
Radiation source: sealed tube	4814 independent reflections
Graphite monochromator	3747 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\rm int} = 0.022$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$\theta_{\max} = 30.0°$, $\theta_{\min} = 2.1°$
$T_{\min} = 0.917$, $T_{\max} = 0.969$	$h = -12\rightarrow12$
	$k = -14\rightarrow14$
	$l = -15\rightarrow15$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.041$	$w = 1/[\sigma^2(F_o^2) + (0.0543P)^2 + 0.1853P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.113$	$(\Delta/\sigma)_{\max} = 0.001$
$S = 1.01$	$\Delta\rho_{\max} = 0.28$ e Å^{-3}
4814 reflections	$\Delta\rho_{\min} = -0.26$ e Å^{-3}
245 parameters	
13 restraints	

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.41790 (14)	0.80484 (13)	0.31353 (12)	0.0397 (2)	
C2	0.30862 (16)	0.67408 (14)	0.29313 (12)	0.0360 (3)	
N3	0.38134 (14)	0.58656 (12)	0.33594 (11)	0.0372 (2)	
C4	0.54988 (17)	0.66946 (15)	0.39106 (13)	0.0375 (3)	
O41	0.64869 (13)	0.62777 (12)	0.44060 (11)	0.0479 (2)	
C5	0.57126 (17)	0.81115 (15)	0.37355 (13)	0.0380 (3)	
C21	0.13212 (16)	0.62953 (15)	0.23374 (13)	0.0382 (3)	
C22	0.0814 (2)	0.75186 (18)	0.23843 (15)	0.0481 (3)	
H22	0.1582	0.8550	0.2805	0.058*	
C23	−0.0814 (2)	0.7210 (2)	0.18129 (19)	0.0597 (4)	
H23	−0.1139	0.8031	0.1846	0.072*	
C24	−0.1961 (2)	0.5686 (2)	0.1193 (2)	0.0640 (5)	
H24	−0.3062	0.5481	0.0818	0.077*	
C25	−0.1476 (2)	0.4469 (2)	0.1131 (2)	0.0650 (5)	
H25	−0.2251	0.3440	0.0702	0.078*	
C26	0.01558 (19)	0.47622 (19)	0.16986 (17)	0.0514 (4)	
H26	0.0472	0.3932	0.1653	0.062*	
N31	0.29845 (15)	0.44083 (12)	0.32625 (12)	0.0389 (2)	
H31	0.3234 (19)	0.4448 (18)	0.4062 (17)	0.047*	
C31	0.30355 (16)	0.30661 (14)	0.21529 (13)	0.0363 (3)	
C32	0.2177 (2)	0.15893 (17)	0.19915 (17)	0.0532 (4)	
H32	0.1654	0.1504	0.2630	0.064*	
C33	0.2098 (3)	0.02409 (19)	0.08817 (19)	0.0666 (5)	
H33	0.1511	−0.0750	0.0775	0.080*	
C34	0.2876 (2)	0.0344 (2)	−0.00687 (17)	0.0620 (4)	
H34	0.2820	−0.0569	−0.0812	0.074*	
C35	0.3728 (2)	0.1801 (2)	0.00926 (16)	0.0584 (4)	
H35	0.4260	0.1877	−0.0544	0.070*	
C36	0.3811 (2)	0.31714 (18)	0.11963 (15)	0.0489 (3)	
H36	0.4387	0.4158	0.1292	0.059*	
C56	0.7166 (2)	0.92665 (15)	0.40655 (13)	0.0395 (3)	0.9005 (19)
H56	0.8101	0.9166	0.4462	0.047*	0.9005 (19)
S51	0.58702 (5)	1.11618 (5)	0.33272 (4)	0.0421 (2)	0.9005 (19)
C52	0.7431 (2)	1.06352 (18)	0.38714 (19)	0.0361 (4)	0.9005 (19)
C53	0.8916 (4)	1.1710 (5)	0.4132 (6)	0.0523 (6)	0.9005 (19)
H53	0.9902	1.1633	0.4457	0.063*	0.9005 (19)
C54	0.8852 (3)	1.2968 (3)	0.3870 (4)	0.0522 (5)	0.9005 (19)
H54	0.9770	1.3778	0.3970	0.063*	0.9005 (19)
C55	0.7272 (2)	1.2830 (2)	0.3453 (3)	0.0497 (4)	0.9005 (19)
H55	0.6978	1.3557	0.3256	0.060*	0.9005 (19)
C66	0.7166 (2)	0.92665 (15)	0.40655 (13)	0.0395 (3)	0.0995 (19)
H66	0.8110	0.9069	0.4284	0.047*	0.0995 (19)
S61	0.9327 (10)	1.1891 (14)	0.4225 (17)	0.0523 (6)	0.0995 (19)
C62	0.7406 (13)	1.0759 (15)	0.411 (2)	0.0361 (4)	0.0995 (19)
C63	0.6243 (15)	1.1204 (18)	0.366 (2)	0.04206 (14)	0.0995 (19)

H63	0.5129	1.0714	0.3527	0.050*	0.0995 (19)
C64	0.6858 (19)	1.248 (2)	0.339 (3)	0.0497 (4)	0.0995 (19)
H64	0.6209	1.2847	0.2966	0.060*	0.0995 (19)
C65	0.8530 (18)	1.311 (3)	0.385 (4)	0.0522 (5)	0.0995 (19)
H65	0.9170	1.4075	0.3941	0.063*	0.0995 (19)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0436 (6)	0.0358 (5)	0.0454 (6)	0.0163 (5)	0.0124 (5)	0.0239 (5)
C2	0.0462 (7)	0.0336 (6)	0.0343 (6)	0.0190 (5)	0.0144 (5)	0.0183 (5)
N3	0.0455 (6)	0.0307 (5)	0.0402 (5)	0.0155 (4)	0.0126 (5)	0.0205 (4)
C4	0.0472 (7)	0.0352 (6)	0.0344 (6)	0.0175 (6)	0.0128 (5)	0.0189 (5)
O41	0.0534 (6)	0.0489 (6)	0.0530 (6)	0.0240 (5)	0.0122 (5)	0.0327 (5)
C5	0.0456 (7)	0.0349 (6)	0.0381 (6)	0.0171 (5)	0.0124 (5)	0.0204 (5)
C21	0.0435 (7)	0.0419 (7)	0.0369 (6)	0.0189 (6)	0.0158 (5)	0.0225 (5)
C22	0.0536 (8)	0.0451 (8)	0.0493 (8)	0.0245 (7)	0.0143 (6)	0.0224 (6)
C23	0.0603 (10)	0.0700 (11)	0.0677 (10)	0.0394 (9)	0.0237 (8)	0.0388 (9)
C24	0.0418 (8)	0.0899 (13)	0.0808 (12)	0.0282 (9)	0.0211 (8)	0.0559 (11)
C25	0.0447 (9)	0.0685 (11)	0.0847 (13)	0.0092 (8)	0.0114 (8)	0.0494 (10)
C26	0.0465 (8)	0.0478 (8)	0.0661 (10)	0.0157 (7)	0.0143 (7)	0.0348 (7)
N31	0.0537 (7)	0.0317 (5)	0.0384 (6)	0.0172 (5)	0.0171 (5)	0.0211 (5)
C31	0.0394 (7)	0.0345 (6)	0.0369 (6)	0.0156 (5)	0.0087 (5)	0.0185 (5)
C32	0.0669 (10)	0.0385 (7)	0.0564 (9)	0.0165 (7)	0.0246 (8)	0.0246 (7)
C33	0.0892 (13)	0.0349 (8)	0.0653 (11)	0.0164 (8)	0.0211 (10)	0.0196 (7)
C34	0.0800 (12)	0.0470 (9)	0.0470 (8)	0.0291 (9)	0.0134 (8)	0.0102 (7)
C35	0.0664 (10)	0.0606 (10)	0.0436 (8)	0.0251 (8)	0.0212 (7)	0.0184 (7)
C36	0.0568 (9)	0.0441 (7)	0.0448 (7)	0.0155 (7)	0.0195 (7)	0.0212 (6)
C56	0.0416 (7)	0.0403 (7)	0.0396 (6)	0.0174 (6)	0.0095 (5)	0.0212 (5)
S51	0.0479 (3)	0.0421 (2)	0.0472 (3)	0.02402 (18)	0.01742 (19)	0.02545 (19)
C52	0.0414 (7)	0.0347 (7)	0.0308 (10)	0.0141 (5)	0.0102 (6)	0.0146 (7)
C53	0.0407 (17)	0.0461 (13)	0.0554 (12)	−0.0009 (13)	0.0075 (18)	0.0241 (12)
C54	0.0605 (12)	0.0340 (8)	0.0524 (9)	0.0067 (8)	0.0166 (11)	0.0190 (7)
C55	0.0698 (14)	0.0323 (11)	0.0527 (9)	0.0202 (10)	0.0234 (11)	0.0232 (10)
C66	0.0416 (7)	0.0403 (7)	0.0396 (6)	0.0174 (6)	0.0095 (5)	0.0212 (5)
S61	0.0407 (17)	0.0461 (13)	0.0554 (12)	−0.0009 (13)	0.0075 (18)	0.0241 (12)
C62	0.0414 (7)	0.0347 (7)	0.0308 (10)	0.0141 (5)	0.0102 (6)	0.0146 (7)
C63	0.0479 (3)	0.0421 (2)	0.0472 (3)	0.02402 (18)	0.01742 (19)	0.02545 (19)
C64	0.0698 (14)	0.0323 (11)	0.0527 (9)	0.0202 (10)	0.0234 (11)	0.0232 (10)
C65	0.0605 (12)	0.0340 (8)	0.0524 (9)	0.0067 (8)	0.0166 (11)	0.0190 (7)

Geometric parameters (Å, º)

N1—C2	1.2996 (16)	C33—C34	1.378 (3)
N1—C5	1.3946 (17)	C33—H33	0.9300
C2—N3	1.4056 (15)	C34—C35	1.364 (2)
C2—C21	1.4643 (19)	C34—H34	0.9300
N3—C4	1.3842 (18)	C35—C36	1.390 (2)

N3—N31	1.3895 (14)	C35—H35	0.9300
C4—O41	1.2225 (15)	C36—H36	0.9300
C4—C5	1.4750 (17)	C56—C52	1.4357 (18)
C5—C56	1.3469 (19)	C56—H56	0.9300
C21—C26	1.389 (2)	S51—C55	1.7197 (17)
C21—C22	1.3977 (18)	S51—C52	1.7233 (18)
C22—C23	1.378 (2)	C52—C53	1.348 (3)
C22—H22	0.9300	C53—C54	1.418 (4)
C23—C24	1.378 (3)	C53—H53	0.9300
C23—H23	0.9300	C54—C55	1.356 (3)
C24—C25	1.376 (3)	C54—H54	0.9300
C24—H24	0.9300	C55—H55	0.9300
C25—C26	1.384 (2)	S61—C62	1.718 (5)
C25—H25	0.9300	S61—C65	1.721 (5)
C26—H26	0.9300	C62—C63	1.343 (6)
N31—C31	1.4216 (16)	C63—C64	1.417 (6)
N31—H31	0.874 (17)	C63—H63	0.9300
C31—C32	1.3825 (19)	C64—C65	1.358 (5)
C31—C36	1.3829 (19)	C64—H64	0.9300
C32—C33	1.381 (2)	C65—H65	0.9300
C32—H32	0.9300		

C2—N1—C5	107.05 (10)	C34—C33—C32	120.83 (16)
N1—C2—N3	111.80 (11)	C34—C33—H33	119.6
N1—C2—C21	122.21 (11)	C32—C33—H33	119.6
N3—C2—C21	125.98 (11)	C35—C34—C33	119.25 (15)
C4—N3—N31	125.00 (10)	C35—C34—H34	120.4
C4—N3—C2	109.11 (10)	C33—C34—H34	120.4
N31—N3—C2	125.89 (11)	C34—C35—C36	120.79 (15)
O41—C4—N3	125.65 (12)	C34—C35—H35	119.6
O41—C4—C5	131.83 (13)	C36—C35—H35	119.6
N3—C4—C5	102.52 (10)	C31—C36—C35	119.90 (14)
C56—C5—N1	125.10 (11)	C31—C36—H36	120.1
C56—C5—C4	125.38 (12)	C35—C36—H36	120.1
N1—C5—C4	109.49 (11)	C5—C56—C52	127.09 (14)
C26—C21—C22	118.77 (13)	C5—C56—H56	116.5
C26—C21—C2	124.94 (12)	C52—C56—H56	116.5
C22—C21—C2	116.25 (12)	C55—S51—C52	91.26 (9)
C23—C22—C21	120.60 (15)	C53—C52—C56	125.2 (2)
C23—C22—H22	119.7	C53—C52—S51	110.74 (19)
C21—C22—H22	119.7	C56—C52—S51	124.05 (13)
C24—C23—C22	120.07 (15)	C52—C53—C54	114.5 (3)
C24—C23—H23	120.0	C52—C53—H53	122.8
C22—C23—H23	120.0	C54—C53—H53	122.8
C25—C24—C23	119.91 (16)	C55—C54—C53	110.88 (18)
C25—C24—H24	120.0	C55—C54—H54	124.6
C23—C24—H24	120.0	C53—C54—H54	124.6
C24—C25—C26	120.59 (17)	C54—C55—S51	112.60 (14)

C24—C25—H25	119.7	C54—C55—H55	123.7
C26—C25—H25	119.7	S51—C55—H55	123.7
C25—C26—C21	120.05 (15)	C62—S61—C65	90.7 (4)
C25—C26—H26	120.0	C63—C62—S61	111.1 (5)
C21—C26—H26	120.0	C62—C63—C64	114.2 (6)
N3—N31—C31	115.70 (10)	C62—C63—H63	122.9
N3—N31—H31	110.4 (11)	C64—C63—H63	122.9
C31—N31—H31	115.2 (10)	C65—C64—C63	110.3 (6)
C32—C31—C36	119.27 (13)	C65—C64—H64	124.9
C32—C31—N31	117.51 (12)	C63—C64—H64	124.9
C36—C31—N31	123.09 (12)	C64—C65—S61	112.1 (6)
C33—C32—C31	119.96 (15)	C64—C65—H65	124.0
C33—C32—H32	120.0	S61—C65—H65	124.0
C31—C32—H32	120.0		

C5—N1—C2—N3	−0.68 (14)	C4—N3—N31—C31	−79.88 (15)
C5—N1—C2—C21	178.68 (11)	C2—N3—N31—C31	100.78 (14)
N1—C2—N3—C4	1.54 (14)	N3—N31—C31—C32	−177.77 (12)
C21—C2—N3—C4	−177.79 (11)	N3—N31—C31—C36	−1.85 (19)
N1—C2—N3—N31	−179.04 (11)	C36—C31—C32—C33	−0.2 (2)
C21—C2—N3—N31	1.63 (19)	N31—C31—C32—C33	175.89 (15)
N31—N3—C4—O41	−1.2 (2)	C31—C32—C33—C34	0.5 (3)
C2—N3—C4—O41	178.22 (12)	C32—C33—C34—C35	−0.2 (3)
N31—N3—C4—C5	178.98 (11)	C33—C34—C35—C36	−0.3 (3)
C2—N3—C4—C5	−1.59 (13)	C32—C31—C36—C35	−0.3 (2)
C2—N1—C5—C56	177.83 (13)	N31—C31—C36—C35	−176.19 (14)
C2—N1—C5—C4	−0.36 (14)	C34—C35—C36—C31	0.6 (3)
O41—C4—C5—C56	3.2 (2)	N1—C5—C56—C52	−0.1 (2)
N3—C4—C5—C56	−176.96 (12)	C4—C5—C56—C52	177.85 (14)
O41—C4—C5—N1	−178.57 (13)	C5—C56—C52—C53	−175.3 (3)
N3—C4—C5—N1	1.22 (13)	C5—C56—C52—S51	7.2 (2)
N1—C2—C21—C26	158.56 (14)	C55—S51—C52—C53	0.4 (3)
N3—C2—C21—C26	−22.2 (2)	C55—S51—C52—C56	178.24 (17)
N1—C2—C21—C22	−18.88 (18)	C56—C52—C53—C54	−179.4 (3)
N3—C2—C21—C22	160.39 (12)	S51—C52—C53—C54	−1.6 (5)
C26—C21—C22—C23	0.3 (2)	C52—C53—C54—C55	2.3 (6)
C2—C21—C22—C23	177.91 (13)	C53—C54—C55—S51	−1.9 (4)
C21—C22—C23—C24	0.3 (2)	C52—S51—C55—C54	0.9 (2)
C22—C23—C24—C25	−0.8 (3)	C65—S61—C62—C63	7 (2)
C23—C24—C25—C26	0.7 (3)	S61—C62—C63—C64	−1 (3)
C24—C25—C26—C21	−0.1 (3)	C62—C63—C64—C65	−8 (3)
C22—C21—C26—C25	−0.4 (2)	C63—C64—C65—S61	13 (4)
C2—C21—C26—C25	−177.77 (14)	C62—S61—C65—C64	−12 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N31—H31···O41^{i}	0.875 (17)	2.136 (18)	2.9767 (17)	161.1 (17)

C34—H34···$Cg1^{ii}$	0.93	2.90	3.654 (2)	140

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

(IV) 3-Anilino-2-(4-methylphenyl)-5-[(thiophen-2-yl)methylidene]-3,5-dihydro-4*H*-imidazol-4-one

Crystal data

$C_{21}H_{17}N_3OS$	$Z = 2$
$M_r = 359.44$	$F(000) = 376$
Triclinic, $P\overline{1}$	$D_x = 1.343$ Mg m^{-3}
$a = 9.1697$ (5) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 10.7939$ (5) Å	Cell parameters from 4395 reflections
$c = 11.0837$ (6) Å	$\theta = 3.7$–28.4°
$\alpha = 66.424$ (2)°	$\mu = 0.20$ mm^{-1}
$\beta = 66.147$ (3)°	$T = 296$ K
$\gamma = 67.934$ (2)°	Block, red
$V = 889.13$ (8) Å^3	0.22 × 0.20 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer	33541 measured reflections
Radiation source: sealed tube	4395 independent reflections
Graphite monochromator	3909 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.023$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$\theta_{max} = 28.4°$, $\theta_{min} = 3.7°$
$T_{min} = 0.911$, $T_{max} = 0.971$	$h = -12 \rightarrow 12$
	$k = -14 \rightarrow 14$
	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.044$	$w = 1/[\sigma^2(F_o^2) + (0.0247P)^2 + 0.7161P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.100$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.14$	$\Delta\rho_{max} = 0.40$ e Å^{-3}
4395 reflections	$\Delta\rho_{min} = -0.28$ e Å^{-3}
255 parameters	
13 restraints	

Special details

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

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

	x	y	z	$U_{iso}*/U_{eq}$	Occ. (<1)
N1	0.24181 (14)	0.75542 (12)	0.76032 (12)	0.0186 (2)	
C2	0.40057 (16)	0.73906 (13)	0.70055 (13)	0.0168 (2)	
N3	0.47389 (13)	0.61663 (12)	0.65903 (12)	0.0174 (2)	
C4	0.35178 (16)	0.55165 (14)	0.69044 (13)	0.0178 (3)	
C5	0.20271 (17)	0.64277 (14)	0.75904 (13)	0.0187 (3)	
O41	0.37406 (12)	0.44584 (10)	0.66276 (10)	0.0214 (2)	

C21	0.48741 (16)	0.83873 (13)	0.67962 (13)	0.0163 (2)	
C22	0.39057 (17)	0.97185 (14)	0.69171 (14)	0.0194 (3)	
H22	0.2766	0.9924	0.7133	0.023*	
C23	0.46361 (18)	1.07287 (14)	0.67168 (14)	0.0216 (3)	
H23	0.3978	1.1606	0.6801	0.026*	
C24	0.63419 (17)	1.04501 (14)	0.63911 (13)	0.0193 (3)	
C25	0.72960 (17)	0.91194 (14)	0.62916 (14)	0.0201 (3)	
H25	0.8435	0.8915	0.6084	0.024*	
C26	0.65865 (17)	0.80934 (14)	0.64956 (14)	0.0192 (3)	
H26	0.7247	0.7211	0.6433	0.023*	
C27	0.71468 (19)	1.15417 (15)	0.61646 (16)	0.0256 (3)	
H27A	0.6328	1.2411	0.6200	0.038*	
H27B	0.7967	1.1664	0.5278	0.038*	
H27C	0.7659	1.1246	0.6872	0.038*	
N31	0.63758 (14)	0.57185 (12)	0.58565 (12)	0.0175 (2)	
H31	0.643 (2)	0.5626 (18)	0.5088 (19)	0.021*	
C31	0.73737 (16)	0.45886 (13)	0.66186 (14)	0.0170 (2)	
C32	0.88742 (16)	0.38721 (14)	0.58827 (14)	0.0202 (3)	
H32	0.9152	0.4101	0.4928	0.024*	
C33	0.99472 (18)	0.28207 (16)	0.65751 (16)	0.0265 (3)	
H33	1.0945	0.2350	0.6079	0.032*	
C34	0.9552 (2)	0.24619 (17)	0.79951 (17)	0.0307 (3)	
H34	1.0277	0.1752	0.8452	0.037*	
C35	0.8063 (2)	0.31706 (17)	0.87319 (16)	0.0297 (3)	
H35	0.7792	0.2934	0.9686	0.036*	
C36	0.69714 (18)	0.42357 (15)	0.80490 (15)	0.0232 (3)	
H36	0.5978	0.4709	0.8547	0.028*	
C56	0.05068 (17)	0.61777 (14)	0.81750 (14)	0.0202 (3)	0.866 (2)
H56	0.0400	0.5388	0.8104	0.024*	0.866 (2)
S51	−0.10269 (6)	0.86039 (5)	0.90082 (6)	0.02351 (14)	0.866 (2)
C52	−0.0945 (10)	0.7026 (17)	0.889 (3)	0.0221 (3)	0.866 (2)
C53	−0.2459 (5)	0.6735 (5)	0.9562 (6)	0.0308 (7)	0.866 (2)
H53	−0.2656	0.5932	0.9604	0.037*	0.866 (2)
C54	−0.3675 (4)	0.7732 (4)	1.0174 (6)	0.0332 (5)	0.866 (2)
H54	−0.4754	0.7669	1.0669	0.040*	0.866 (2)
C55	−0.3082 (3)	0.8815 (4)	0.9956 (5)	0.0289 (5)	0.866 (2)
H55	−0.3717	0.9584	1.0282	0.035*	0.866 (2)
C66	0.05068 (17)	0.61777 (14)	0.81750 (14)	0.0202 (3)	0.134 (2)
H66	0.0405	0.5390	0.8095	0.024*	0.134 (2)
S61	−0.2796 (10)	0.6522 (9)	0.9676 (10)	0.0308 (7)	0.134 (2)
C62	−0.096 (6)	0.701 (11)	0.890 (17)	0.0221 (3)	0.134 (2)
C63	−0.1170 (16)	0.8237 (14)	0.9132 (18)	0.02351 (14)	0.134 (2)
H63	−0.0310	0.8653	0.8822	0.028*	0.134 (2)
C64	−0.276 (2)	0.881 (3)	0.986 (4)	0.0289 (5)	0.134 (2)
H64	−0.3089	0.9651	1.0066	0.035*	0.134 (2)
C65	−0.378 (2)	0.798 (3)	1.023 (5)	0.0332 (5)	0.134 (2)
H65	−0.4886	0.8190	1.0733	0.040*	0.134 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0192 (5)	0.0186 (5)	0.0191 (5)	−0.0041 (4)	−0.0072 (4)	−0.0056 (4)
C2	0.0195 (6)	0.0158 (6)	0.0165 (6)	−0.0016 (5)	−0.0084 (5)	−0.0057 (5)
N3	0.0169 (5)	0.0164 (5)	0.0211 (5)	−0.0023 (4)	−0.0068 (4)	−0.0084 (4)
C4	0.0205 (6)	0.0173 (6)	0.0168 (6)	−0.0051 (5)	−0.0085 (5)	−0.0029 (5)
C5	0.0212 (6)	0.0175 (6)	0.0178 (6)	−0.0030 (5)	−0.0083 (5)	−0.0047 (5)
O41	0.0274 (5)	0.0180 (5)	0.0219 (5)	−0.0065 (4)	−0.0088 (4)	−0.0068 (4)
C21	0.0190 (6)	0.0154 (6)	0.0167 (6)	−0.0029 (5)	−0.0078 (5)	−0.0056 (4)
C22	0.0185 (6)	0.0183 (6)	0.0209 (6)	−0.0015 (5)	−0.0072 (5)	−0.0069 (5)
C23	0.0260 (7)	0.0143 (6)	0.0239 (7)	−0.0014 (5)	−0.0096 (5)	−0.0064 (5)
C24	0.0263 (7)	0.0171 (6)	0.0170 (6)	−0.0066 (5)	−0.0098 (5)	−0.0029 (5)
C25	0.0200 (6)	0.0198 (6)	0.0229 (6)	−0.0043 (5)	−0.0094 (5)	−0.0064 (5)
C26	0.0202 (6)	0.0168 (6)	0.0225 (6)	−0.0015 (5)	−0.0087 (5)	−0.0081 (5)
C27	0.0322 (8)	0.0194 (6)	0.0294 (7)	−0.0092 (6)	−0.0136 (6)	−0.0043 (6)
N31	0.0170 (5)	0.0179 (5)	0.0177 (5)	−0.0014 (4)	−0.0050 (4)	−0.0082 (4)
C31	0.0180 (6)	0.0152 (6)	0.0222 (6)	−0.0057 (5)	−0.0071 (5)	−0.0072 (5)
C32	0.0193 (6)	0.0203 (6)	0.0222 (6)	−0.0061 (5)	−0.0037 (5)	−0.0087 (5)
C33	0.0202 (7)	0.0254 (7)	0.0335 (8)	−0.0002 (5)	−0.0082 (6)	−0.0133 (6)
C34	0.0286 (8)	0.0278 (8)	0.0360 (8)	0.0037 (6)	−0.0195 (7)	−0.0099 (6)
C35	0.0347 (8)	0.0304 (8)	0.0233 (7)	−0.0025 (6)	−0.0137 (6)	−0.0074 (6)
C36	0.0222 (6)	0.0242 (7)	0.0221 (7)	−0.0017 (5)	−0.0060 (5)	−0.0101 (5)
C56	0.0224 (6)	0.0197 (6)	0.0194 (6)	−0.0056 (5)	−0.0104 (5)	−0.0022 (5)
S51	0.0200 (2)	0.0245 (3)	0.0267 (2)	0.00036 (17)	−0.01011 (16)	−0.0106 (2)
C52	0.0211 (7)	0.0244 (8)	0.0189 (8)	−0.0047 (5)	−0.0097 (5)	−0.0017 (7)
C53	0.032 (2)	0.0277 (16)	0.0293 (11)	−0.0071 (10)	−0.0133 (14)	−0.0016 (11)
C54	0.0227 (8)	0.0347 (18)	0.0292 (10)	−0.0041 (10)	−0.0061 (7)	−0.0016 (14)
C55	0.0160 (11)	0.0365 (9)	0.0264 (11)	0.0040 (10)	−0.0057 (13)	−0.0122 (7)
C66	0.0224 (6)	0.0197 (6)	0.0194 (6)	−0.0056 (5)	−0.0104 (5)	−0.0022 (5)
S61	0.032 (2)	0.0277 (16)	0.0293 (11)	−0.0071 (10)	−0.0133 (14)	−0.0016 (11)
C62	0.0211 (7)	0.0244 (8)	0.0189 (8)	−0.0047 (5)	−0.0097 (5)	−0.0017 (7)
C63	0.0200 (2)	0.0245 (3)	0.0267 (2)	0.00036 (17)	−0.01011 (16)	−0.0106 (2)
C64	0.0160 (11)	0.0365 (9)	0.0264 (11)	0.0040 (10)	−0.0057 (13)	−0.0122 (7)
C65	0.0227 (8)	0.0347 (18)	0.0292 (10)	−0.0041 (10)	−0.0061 (7)	−0.0016 (14)

Geometric parameters (Å, º)

N1—C2	1.3075 (17)	C32—C33	1.386 (2)
N1—C5	1.3974 (17)	C32—H32	0.9300
C2—N3	1.4067 (16)	C33—C34	1.384 (2)
C2—C21	1.4628 (18)	C33—H33	0.9300
N3—N31	1.3893 (15)	C34—C35	1.389 (2)
N3—C4	1.3952 (17)	C34—H34	0.9300
C4—O41	1.2225 (16)	C35—C36	1.396 (2)
C4—C5	1.4702 (18)	C35—H35	0.9300
C5—C56	1.3595 (19)	C36—H36	0.9300
C21—C26	1.4016 (18)	C56—C52	1.433 (2)

C21—C22	1.4037 (18)	C56—H56	0.9300
C22—C23	1.3861 (19)	S51—C55	1.724 (2)
C22—H22	0.9300	S51—C52	1.730 (3)
C23—C24	1.394 (2)	C52—C53	1.374 (4)
C23—H23	0.9300	C53—C54	1.390 (5)
C24—C25	1.3947 (19)	C53—H53	0.9300
C24—C27	1.5043 (19)	C54—C55	1.367 (3)
C25—C26	1.3867 (19)	C54—H54	0.9300
C25—H25	0.9300	C55—H55	0.9300
C26—H26	0.9300	S61—C65	1.715 (10)
C27—H27A	0.9600	S61—C62	1.731 (11)
C27—H27B	0.9600	C62—C63	1.374 (10)
C27—H27C	0.9600	C63—C64	1.391 (10)
N31—C31	1.4144 (17)	C63—H63	0.9300
N31—H31	0.876 (18)	C64—C65	1.363 (9)
C31—C36	1.3944 (19)	C64—H64	0.9300
C31—C32	1.3979 (18)	C65—H65	0.9300

C2—N1—C5	106.80 (11)	C33—C32—C31	120.00 (13)
N1—C2—N3	111.74 (11)	C33—C32—H32	120.0
N1—C2—C21	122.93 (12)	C31—C32—H32	120.0
N3—C2—C21	125.32 (12)	C34—C33—C32	120.84 (14)
N31—N3—C4	123.73 (11)	C34—C33—H33	119.6
N31—N3—C2	126.90 (11)	C32—C33—H33	119.6
C4—N3—C2	109.06 (11)	C33—C34—C35	119.43 (14)
O41—C4—N3	125.35 (12)	C33—C34—H34	120.3
O41—C4—C5	132.29 (13)	C35—C34—H34	120.3
N3—C4—C5	102.35 (11)	C34—C35—C36	120.43 (14)
C56—C5—N1	125.10 (13)	C34—C35—H35	119.8
C56—C5—C4	124.86 (13)	C36—C35—H35	119.8
N1—C5—C4	109.99 (11)	C31—C36—C35	119.87 (13)
C26—C21—C22	118.71 (12)	C31—C36—H36	120.1
C26—C21—C2	124.37 (12)	C35—C36—H36	120.1
C22—C21—C2	116.92 (12)	C5—C56—C52	125.57 (18)
C23—C22—C21	120.48 (12)	C5—C56—H56	117.2
C23—C22—H22	119.8	C52—C56—H56	117.2
C21—C22—H22	119.8	C55—S51—C52	90.99 (12)
C22—C23—C24	121.02 (12)	C53—C52—C56	126.0 (3)
C22—C23—H23	119.5	C53—C52—S51	110.2 (2)
C24—C23—H23	119.5	C56—C52—S51	123.8 (2)
C23—C24—C25	118.25 (12)	C52—C53—C54	114.9 (3)
C23—C24—C27	121.42 (13)	C52—C53—H53	122.6
C25—C24—C27	120.33 (13)	C54—C53—H53	122.6
C26—C25—C24	121.54 (13)	C55—C54—C53	111.2 (2)
C26—C25—H25	119.2	C55—C54—H54	124.4
C24—C25—H25	119.2	C53—C54—H54	124.4
C25—C26—C21	119.98 (12)	C54—C55—S51	112.7 (2)
C25—C26—H26	120.0	C54—C55—H55	123.6

C21—C26—H26	120.0	S51—C55—H55	123.6
C24—C27—H27A	109.5	C65—S61—C62	91.3 (6)
C24—C27—H27B	109.5	C63—C62—S61	109.4 (6)
H27A—C27—H27B	109.5	C62—C63—C64	115.4 (9)
C24—C27—H27C	109.5	C62—C63—H63	122.3
H27A—C27—H27C	109.5	C64—C63—H63	122.3
H27B—C27—H27C	109.5	C65—C64—C63	110.8 (11)
N3—N31—C31	116.19 (11)	C65—C64—H64	124.6
N3—N31—H31	110.1 (11)	C63—C64—H64	124.6
C31—N31—H31	115.0 (11)	C64—C65—S61	113.0 (10)
C36—C31—C32	119.44 (12)	C64—C65—H65	123.5
C36—C31—N31	122.61 (12)	S61—C65—H65	123.5
C32—C31—N31	117.79 (12)		

C5—N1—C2—N3	−0.76 (14)	C2—C21—C26—C25	178.90 (12)
C5—N1—C2—C21	178.62 (11)	C4—N3—N31—C31	−81.40 (15)
N1—C2—N3—N31	175.90 (11)	C2—N3—N31—C31	105.69 (14)
C21—C2—N3—N31	−3.5 (2)	N3—N31—C31—C36	−21.06 (18)
N1—C2—N3—C4	2.13 (15)	N3—N31—C31—C32	163.55 (11)
C21—C2—N3—C4	−177.23 (12)	C36—C31—C32—C33	0.0 (2)
N31—N3—C4—O41	3.0 (2)	N31—C31—C32—C33	175.57 (12)
C2—N3—C4—O41	177.05 (12)	C31—C32—C33—C34	0.2 (2)
N31—N3—C4—C5	−176.39 (11)	C32—C33—C34—C35	−0.3 (2)
C2—N3—C4—C5	−2.39 (13)	C33—C34—C35—C36	0.0 (3)
C2—N1—C5—C56	176.70 (13)	C32—C31—C36—C35	−0.2 (2)
C2—N1—C5—C4	−0.81 (14)	N31—C31—C36—C35	−175.57 (13)
O41—C4—C5—C56	5.1 (2)	C34—C35—C36—C31	0.2 (2)
N3—C4—C5—C56	−175.52 (13)	N1—C5—C56—C52	0.4 (15)
O41—C4—C5—N1	−177.38 (14)	C4—C5—C56—C52	177.5 (15)
N3—C4—C5—N1	2.00 (14)	C5—C56—C52—C53	−175.5 (16)
N1—C2—C21—C26	162.87 (13)	C5—C56—C52—S51	5 (3)
N3—C2—C21—C26	−17.8 (2)	C55—S51—C52—C53	0.2 (18)
N1—C2—C21—C22	−16.72 (18)	C55—S51—C52—C56	179 (2)
N3—C2—C21—C22	162.58 (12)	C56—C52—C53—C54	−179.7 (19)
C26—C21—C22—C23	1.2 (2)	S51—C52—C53—C54	−1 (2)
C2—C21—C22—C23	−179.19 (12)	C52—C53—C54—C55	0.6 (16)
C21—C22—C23—C24	0.0 (2)	C53—C54—C55—S51	−0.4 (7)
C22—C23—C24—C25	−0.9 (2)	C52—S51—C55—C54	0.1 (11)
C22—C23—C24—C27	179.68 (13)	C65—S61—C62—C63	1 (11)
C23—C24—C25—C26	0.6 (2)	S61—C62—C63—C64	−2 (14)
C27—C24—C25—C26	179.99 (13)	C62—C63—C64—C65	2 (10)
C24—C25—C26—C21	0.6 (2)	C63—C64—C65—S61	−1 (5)
C22—C21—C26—C25	−1.53 (19)	C62—S61—C65—C64	0 (8)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N31—H31···O41^{i}	0.876 (19)	2.010 (19)	2.8813 (16)	172.9 (19)

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

(V) 2-Phenyl-5-[(thiophen-2-yl)methylidene]-3-{[(*E*)-(thiophen-2-yl)methylidene]amino}-3,5-dihydro-4*H*-imidazol-4-one

Crystal data

$C_{19}H_{13}N_3OS_2$	D_x = 1.432 Mg m^{-3}
M_r = 363.44	Mo $K\alpha$ radiation, λ = 0.71073 Å
Orthorhombic, *Pbca*	Cell parameters from 6527 reflections
a = 8.2717 (2) Å	θ = 3.2–33.4°
b = 17.4530 (3) Å	μ = 0.33 mm^{-1}
c = 23.3501 (5) Å	T = 296 K
V = 3370.96 (12) Å^3	Block, brown
Z = 8	0.45 × 0.45 × 0.45 mm
F(000) = 1504	

Data collection

Bruker APEXII CCD diffractometer	24050 measured reflections
Radiation source: sealed tube	4912 independent reflections
Graphite monochromator	3966 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.021
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	θ_{max} = 30.0°, θ_{min} = 3.2°
T_{min} = 0.817, T_{max} = 0.863	$h = -11\rightarrow10$
	$k = -23\rightarrow24$
	$l = -32\rightarrow29$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.043	$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 1.3618P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.120	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.02	$\Delta\rho_{max}$ = 0.33 e Å^{-3}
4912 reflections	$\Delta\rho_{min}$ = −0.33 e Å^{-3}
242 parameters	
13 restraints	

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.54252 (15)	0.55168 (7)	0.56565 (5)	0.0371 (3)	
C2	0.51062 (16)	0.49658 (7)	0.60166 (6)	0.0337 (3)	
N3	0.63071 (15)	0.44002 (7)	0.60204 (5)	0.0381 (3)	
C4	0.75032 (18)	0.46075 (8)	0.56229 (7)	0.0418 (3)	
O41	0.87160 (14)	0.42511 (7)	0.55057 (6)	0.0605 (4)	
C5	0.68887 (18)	0.53423 (8)	0.54010 (6)	0.0383 (3)	

C21	0.35853 (16)	0.49441 (7)	0.63427 (6)	0.0339 (3)	
C22	0.2369 (2)	0.54397 (9)	0.61536 (7)	0.0435 (3)	
H22	0.2562	0.5761	0.5844	0.052*	
C23	0.0887 (2)	0.54564 (10)	0.64220 (8)	0.0519 (4)	
H23	0.0085	0.5786	0.6291	0.062*	
C24	0.0588 (2)	0.49878 (10)	0.68825 (8)	0.0521 (4)	
H24	−0.0416	0.4998	0.7062	0.063*	
C25	0.1781 (2)	0.45034 (10)	0.70780 (7)	0.0531 (4)	
H25	0.1582	0.4192	0.7394	0.064*	
C26	0.3275 (2)	0.44748 (9)	0.68100 (6)	0.0445 (3)	
H26	0.4068	0.4142	0.6943	0.053*	
N31	0.63111 (15)	0.37886 (7)	0.63997 (5)	0.0402 (3)	
C36	0.7208 (2)	0.32114 (8)	0.62697 (7)	0.0435 (3)	
H36	0.7806	0.3217	0.5933	0.052*	
S31	0.61844 (7)	0.24869 (3)	0.72607 (2)	0.06214 (16)	
C32	0.72870 (19)	0.25580 (8)	0.66436 (7)	0.0412 (3)	
C33	0.8194 (2)	0.19123 (9)	0.65582 (8)	0.0496 (4)	
H33	0.8870	0.1841	0.6245	0.060*	
C34	0.7991 (2)	0.13664 (9)	0.69970 (8)	0.0519 (4)	
H34	0.8520	0.0896	0.7005	0.062*	
C35	0.6957 (2)	0.15988 (10)	0.73985 (8)	0.0561 (4)	
H35	0.6686	0.1309	0.7719	0.067*	
C56	0.76479 (18)	0.57556 (8)	0.49951 (6)	0.0392 (3)	0.9763 (18)
H56	0.8624	0.5560	0.4862	0.047*	0.9763 (18)
S51	0.54739 (6)	0.69862 (3)	0.49768 (2)	0.0496 (2)	0.9763 (18)
C52	0.71238 (18)	0.64605 (9)	0.47452 (9)	0.0376 (4)	0.9763 (18)
C53	0.7855 (3)	0.68004 (13)	0.42842 (10)	0.0480 (4)	0.9763 (18)
H53	0.8759	0.6603	0.4099	0.058*	0.9763 (18)
C54	0.7063 (2)	0.74942 (10)	0.41224 (8)	0.0547 (4)	0.9763 (18)
H54	0.7382	0.7796	0.3814	0.066*	0.9763 (18)
C55	0.5796 (3)	0.76645 (11)	0.44661 (8)	0.0556 (5)	0.9763 (18)
H55	0.5170	0.8104	0.4428	0.067*	0.9763 (18)
C66	0.76479 (18)	0.57556 (8)	0.49951 (6)	0.0392 (3)	0.0237 (18)
H66	0.8693	0.5630	0.4883	0.047*	0.0237 (18)
S61	0.761 (4)	0.6994 (14)	0.4220 (12)	0.0480 (4)	0.0237 (18)
C62	0.683 (6)	0.639 (4)	0.474 (3)	0.0376 (4)	0.0237 (18)
C63	0.531 (4)	0.662 (3)	0.490 (3)	0.04958 (15)	0.0237 (18)
H63	0.4569	0.6327	0.5101	0.059*	0.0237 (18)
C64	0.504 (6)	0.740 (2)	0.472 (2)	0.0556 (5)	0.0237 (18)
H64	0.4302	0.7729	0.4886	0.067*	0.0237 (18)
C65	0.598 (7)	0.758 (3)	0.426 (3)	0.0547 (4)	0.0237 (18)
H65	0.5756	0.7974	0.4005	0.066*	0.0237 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0380 (6)	0.0322 (5)	0.0411 (6)	−0.0013 (5)	0.0031 (5)	0.0017 (4)
C2	0.0350 (6)	0.0295 (6)	0.0366 (6)	−0.0011 (5)	0.0004 (5)	−0.0032 (5)

N3	0.0371 (6)	0.0324 (5)	0.0447 (6)	0.0010 (5)	0.0040 (5)	0.0036 (5)
C4	0.0360 (7)	0.0372 (7)	0.0522 (8)	−0.0024 (6)	0.0060 (6)	0.0022 (6)
O41	0.0416 (6)	0.0473 (6)	0.0926 (10)	0.0093 (5)	0.0222 (6)	0.0159 (6)
C5	0.0369 (7)	0.0334 (6)	0.0447 (7)	−0.0031 (5)	0.0016 (6)	−0.0007 (5)
C21	0.0368 (6)	0.0304 (6)	0.0344 (6)	−0.0018 (5)	0.0030 (5)	−0.0051 (5)
C22	0.0476 (8)	0.0380 (7)	0.0449 (8)	0.0043 (6)	0.0074 (7)	0.0054 (6)
C23	0.0457 (8)	0.0516 (9)	0.0583 (9)	0.0124 (7)	0.0085 (7)	0.0066 (7)
C24	0.0455 (8)	0.0541 (9)	0.0568 (9)	0.0032 (7)	0.0169 (7)	0.0019 (7)
C25	0.0585 (10)	0.0527 (9)	0.0481 (8)	0.0032 (8)	0.0181 (8)	0.0118 (7)
C26	0.0473 (8)	0.0451 (8)	0.0410 (7)	0.0076 (6)	0.0052 (6)	0.0062 (6)
N31	0.0437 (6)	0.0310 (5)	0.0458 (6)	0.0001 (5)	0.0011 (5)	0.0055 (5)
C36	0.0484 (8)	0.0369 (7)	0.0451 (7)	0.0028 (6)	0.0057 (6)	0.0024 (6)
S31	0.0729 (3)	0.0491 (2)	0.0644 (3)	0.0114 (2)	0.0236 (2)	0.0113 (2)
C32	0.0441 (7)	0.0348 (7)	0.0448 (7)	0.0009 (6)	0.0016 (6)	0.0023 (6)
C33	0.0553 (9)	0.0413 (8)	0.0522 (9)	0.0081 (7)	0.0063 (7)	0.0030 (7)
C34	0.0547 (9)	0.0364 (7)	0.0645 (10)	0.0055 (7)	−0.0056 (8)	0.0069 (7)
C35	0.0652 (11)	0.0453 (8)	0.0578 (10)	−0.0023 (8)	0.0001 (9)	0.0157 (7)
C56	0.0363 (7)	0.0354 (7)	0.0458 (7)	−0.0033 (5)	0.0037 (6)	−0.0012 (6)
S51	0.0520 (2)	0.0488 (3)	0.0480 (2)	0.01105 (18)	0.00475 (18)	0.00530 (17)
C52	0.0382 (8)	0.0343 (7)	0.0402 (7)	−0.0054 (6)	0.0000 (7)	−0.0019 (6)
C53	0.0522 (11)	0.0368 (11)	0.0549 (10)	−0.0043 (8)	0.0021 (8)	0.0057 (8)
C54	0.0653 (11)	0.0440 (9)	0.0549 (10)	−0.0082 (8)	−0.0003 (9)	0.0131 (7)
C55	0.0649 (11)	0.0448 (9)	0.0570 (11)	0.0071 (8)	−0.0069 (10)	0.0097 (8)
C66	0.0363 (7)	0.0354 (7)	0.0458 (7)	−0.0033 (5)	0.0037 (6)	−0.0012 (6)
S61	0.0522 (11)	0.0368 (11)	0.0549 (10)	−0.0043 (8)	0.0021 (8)	0.0057 (8)
C62	0.0382 (8)	0.0343 (7)	0.0402 (7)	−0.0054 (6)	0.0000 (7)	−0.0019 (6)
C63	0.0520 (2)	0.0488 (3)	0.0480 (2)	0.01105 (18)	0.00475 (18)	0.00530 (17)
C64	0.0649 (11)	0.0448 (9)	0.0570 (11)	0.0071 (8)	−0.0069 (10)	0.0097 (8)
C65	0.0653 (11)	0.0440 (9)	0.0549 (10)	−0.0082 (8)	−0.0003 (9)	0.0131 (7)

Geometric parameters (Å, º)

N1—C2	1.3043 (17)	C32—C33	1.368 (2)
N1—C5	1.3836 (19)	C33—C34	1.409 (2)
C2—N3	1.4004 (17)	C33—H33	0.9300
C2—C21	1.4710 (19)	C34—C35	1.332 (3)
N3—N31	1.3872 (16)	C34—H34	0.9300
N3—C4	1.4040 (19)	C35—H35	0.9300
C4—O41	1.2117 (18)	C56—C52	1.429 (2)
C4—C5	1.473 (2)	C56—H56	0.9300
C5—C56	1.347 (2)	S51—C55	1.701 (2)
C21—C26	1.388 (2)	S51—C52	1.7311 (18)
C21—C22	1.399 (2)	C52—C53	1.370 (3)
C22—C23	1.377 (2)	C53—C54	1.428 (3)
C22—H22	0.9300	C53—H53	0.9300
C23—C24	1.373 (2)	C54—C55	1.353 (3)
C23—H23	0.9300	C54—H54	0.9300
C24—C25	1.378 (3)	C55—H55	0.9300

C24—H24	0.9300	S61—C65	1.699 (10)
C25—C26	1.386 (2)	S61—C62	1.732 (10)
C25—H25	0.9300	C62—C63	1.374 (10)
C26—H26	0.9300	C63—C64	1.431 (10)
N31—C36	1.2872 (19)	C63—H63	0.9300
C36—C32	1.438 (2)	C64—C65	1.355 (10)
C36—H36	0.9300	C64—H64	0.9300
S31—C35	1.7071 (18)	C65—H65	0.9300
S31—C32	1.7099 (16)		

C2—N1—C5	107.02 (12)	C36—C32—S31	123.03 (11)
N1—C2—N3	112.35 (12)	C32—C33—C34	112.68 (15)
N1—C2—C21	121.71 (12)	C32—C33—H33	123.7
N3—C2—C21	125.82 (12)	C34—C33—H33	123.7
N31—N3—C2	123.24 (12)	C35—C34—C33	112.48 (15)
N31—N3—C4	128.23 (12)	C35—C34—H34	123.8
C2—N3—C4	108.30 (11)	C33—C34—H34	123.8
O41—C4—N3	126.90 (14)	C34—C35—S31	112.59 (13)
O41—C4—C5	130.77 (14)	C34—C35—H35	123.7
N3—C4—C5	102.33 (12)	S31—C35—H35	123.7
C56—C5—N1	126.42 (14)	C5—C56—C52	127.39 (15)
C56—C5—C4	123.57 (14)	C5—C56—H56	116.3
N1—C5—C4	109.98 (12)	C52—C56—H56	116.3
C26—C21—C22	118.68 (13)	C55—S51—C52	91.51 (9)
C26—C21—C2	125.46 (13)	C53—C52—C56	124.03 (17)
C22—C21—C2	115.85 (12)	C53—C52—S51	111.36 (14)
C23—C22—C21	120.66 (14)	C56—C52—S51	124.60 (12)
C23—C22—H22	119.7	C52—C53—C54	111.86 (19)
C21—C22—H22	119.7	C52—C53—H53	124.1
C24—C23—C22	120.26 (16)	C54—C53—H53	124.1
C24—C23—H23	119.9	C55—C54—C53	112.64 (17)
C22—C23—H23	119.9	C55—C54—H54	123.7
C23—C24—C25	119.74 (15)	C53—C54—H54	123.7
C23—C24—H24	120.1	C54—C55—S51	112.59 (14)
C25—C24—H24	120.1	C54—C55—H55	123.7
C24—C25—C26	120.74 (15)	S51—C55—H55	123.7
C24—C25—H25	119.6	C65—S61—C62	91.6 (7)
C26—C25—H25	119.6	C63—C62—S61	110.6 (9)
C25—C26—C21	119.91 (15)	C62—C63—C64	110.1 (14)
C25—C26—H26	120.0	C62—C63—H63	124.9
C21—C26—H26	120.0	C64—C63—H63	124.9
C36—N31—N3	116.94 (13)	C65—C64—C63	111.5 (14)
N31—C36—C32	120.26 (14)	C65—C64—H64	124.2
N31—C36—H36	119.9	C63—C64—H64	124.2
C32—C36—H36	119.9	C64—C65—S61	111.1 (12)
C35—S31—C32	91.44 (8)	C64—C65—H65	124.5
C33—C32—C36	126.15 (15)	S61—C65—H65	124.5
C33—C32—S31	110.81 (12)		

C5—N1—C2—N3	−0.36 (16)	C2—N3—N31—C36	162.33 (14)
C5—N1—C2—C21	−176.58 (12)	C4—N3—N31—C36	−23.9 (2)
N1—C2—N3—N31	174.56 (12)	N3—N31—C36—C32	179.49 (13)
C21—C2—N3—N31	−9.4 (2)	N31—C36—C32—C33	−179.62 (17)
N1—C2—N3—C4	−0.32 (16)	N31—C36—C32—S31	1.9 (2)
C21—C2—N3—C4	175.71 (13)	C35—S31—C32—C33	0.32 (14)
N31—N3—C4—O41	6.4 (3)	C35—S31—C32—C36	178.98 (15)
C2—N3—C4—O41	−179.10 (17)	C36—C32—C33—C34	−178.92 (16)
N31—N3—C4—C5	−173.76 (13)	S31—C32—C33—C34	−0.3 (2)
C2—N3—C4—C5	0.80 (15)	C32—C33—C34—C35	0.1 (2)
C2—N1—C5—C56	178.84 (15)	C33—C34—C35—S31	0.1 (2)
C2—N1—C5—C4	0.88 (16)	C32—S31—C35—C34	−0.25 (16)
O41—C4—C5—C56	0.8 (3)	N1—C5—C56—C52	0.6 (3)
N3—C4—C5—C56	−179.06 (14)	C4—C5—C56—C52	178.29 (16)
O41—C4—C5—N1	178.85 (18)	C5—C56—C52—C53	−171.51 (19)
N3—C4—C5—N1	−1.03 (16)	C5—C56—C52—S51	7.5 (3)
N1—C2—C21—C26	−166.60 (14)	C55—S51—C52—C53	−1.11 (18)
N3—C2—C21—C26	17.7 (2)	C55—S51—C52—C56	179.79 (17)
N1—C2—C21—C22	13.90 (19)	C56—C52—C53—C54	179.28 (18)
N3—C2—C21—C22	−161.79 (14)	S51—C52—C53—C54	0.2 (2)
C26—C21—C22—C23	−0.6 (2)	C52—C53—C54—C55	1.2 (3)
C2—C21—C22—C23	178.89 (15)	C53—C54—C55—S51	−2.1 (2)
C21—C22—C23—C24	0.4 (3)	C52—S51—C55—C54	1.82 (17)
C22—C23—C24—C25	0.4 (3)	C65—S61—C62—C63	4 (7)
C23—C24—C25—C26	−1.0 (3)	S61—C62—C63—C64	−16 (8)
C24—C25—C26—C21	0.7 (3)	C62—C63—C64—C65	24 (7)
C22—C21—C26—C25	0.1 (2)	C63—C64—C65—S61	−22 (7)
C2—C21—C26—C25	−179.38 (15)	C62—S61—C65—C64	10 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C56—H56···O41^{i}	0.93	2.38	3.2270 (19)	151
C35—H35···*Cg*2^{ii}	0.93	2.99	3.7477 (19)	149

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

(VI) Methyl (2*Z*)-2-(benzoylamino)-3-(thiophen-2-yl)prop-2-enoate

Crystal data

$C_{15}H_{13}NO_3S$
$M_r = 287.32$
Orthorhombic, *Pbca*
$a = 16.6123$ (3) Å
$b = 9.7589$ (2) Å
$c = 17.7543$ (4) Å
$V = 2878.29$ (10) Å^3
$Z = 8$
$F(000) = 1200$

$D_x = 1.326$ Mg m^{-3}
Mo *Kα* radiation, $\lambda = 0.71073$ Å
Cell parameters from 5464 reflections
$\theta = 2.3$–33.3°
$\mu = 0.23$ mm^{-1}
$T = 296$ K
Plate, yellow
0.45 × 0.25 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer	18087 measured reflections
Radiation source: sealed tube	3292 independent reflections
Graphite monochromator	2439 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.023$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$\theta_{\max} = 27.5°$, $\theta_{\min} = 2.3°$
$T_{\min} = 0.881$, $T_{\max} = 0.966$	$h = -21 \rightarrow 21$
	$k = -12 \rightarrow 10$
	$l = -23 \rightarrow 20$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.045$	$w = 1/[\sigma^2(F_o^2) + (0.042P)^2 + 1.4284P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.118$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.07$	$\Delta\rho_{\max} = 0.27$ e Å^{-3}
3292 reflections	$\Delta\rho_{\min} = -0.40$ e Å^{-3}
185 parameters	
0 restraints	

Special details

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

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

	x	y	z	U_{iso}*/U_{eq}
C1	0.39384 (11)	0.62770 (18)	0.53738 (11)	0.0442 (4)
C2	0.31535 (10)	0.57995 (17)	0.50811 (10)	0.0384 (4)
C3	0.29934 (11)	0.5682 (2)	0.43467 (10)	0.0455 (4)
H3	0.3406	0.5950	0.4024	0.055*
O1	0.40936 (9)	0.63862 (17)	0.60332 (8)	0.0641 (4)
O2	0.44645 (8)	0.65538 (15)	0.48255 (8)	0.0564 (4)
C4	0.52373 (13)	0.7078 (3)	0.50626 (16)	0.0749 (7)
H4A	0.5486	0.6437	0.5400	0.112*
H4B	0.5164	0.7939	0.5315	0.112*
H4C	0.5575	0.7208	0.4630	0.112*
N21	0.25788 (9)	0.54444 (15)	0.56377 (8)	0.0385 (3)
H21	0.2558 (13)	0.464 (2)	0.5794 (11)	0.046*
C27	0.21473 (10)	0.64027 (17)	0.60024 (10)	0.0381 (4)
O27	0.22034 (9)	0.76190 (13)	0.58389 (9)	0.0590 (4)
C21	0.15897 (11)	0.59269 (19)	0.66080 (10)	0.0409 (4)
C22	0.13345 (11)	0.4583 (2)	0.66694 (11)	0.0459 (4)
H22	0.1503	0.3934	0.6319	0.055*
C23	0.08261 (13)	0.4202 (3)	0.72545 (13)	0.0627 (6)
H23	0.0649	0.3300	0.7291	0.075*
C24	0.05840 (13)	0.5149 (3)	0.77779 (13)	0.0712 (7)
H24	0.0251	0.4885	0.8173	0.085*

C25	0.08327 (16)	0.6481 (3)	0.77185 (13)	0.0749 (8)
H25	0.0667	0.7123	0.8073	0.090*
C26	0.13294 (14)	0.6876 (2)	0.71336 (12)	0.0609 (6)
H26	0.1490	0.7786	0.7093	0.073*
S31	0.14184 (3)	0.46353 (6)	0.44381 (3)	0.05686 (18)
C32	0.22727 (11)	0.5198 (2)	0.39847 (11)	0.0468 (4)
C33	0.21761 (13)	0.5075 (3)	0.32222 (12)	0.0665 (6)
H33	0.2572	0.5320	0.2877	0.080*
C34	0.14263 (14)	0.4544 (3)	0.30099 (14)	0.0694 (7)
H34	0.1270	0.4403	0.2513	0.083*
C35	0.09598 (14)	0.4264 (3)	0.36063 (13)	0.0657 (6)
H35	0.0443	0.3905	0.3569	0.079*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0444 (10)	0.0339 (9)	0.0542 (11)	0.0028 (8)	−0.0018 (8)	0.0021 (8)
C2	0.0400 (9)	0.0312 (8)	0.0440 (9)	0.0007 (7)	0.0028 (7)	0.0025 (7)
C3	0.0388 (9)	0.0513 (11)	0.0465 (10)	−0.0001 (8)	0.0046 (8)	0.0021 (9)
O1	0.0583 (9)	0.0797 (11)	0.0541 (8)	−0.0039 (8)	−0.0104 (7)	−0.0067 (8)
O2	0.0411 (7)	0.0644 (9)	0.0637 (9)	−0.0106 (7)	−0.0007 (6)	0.0089 (7)
C4	0.0450 (11)	0.0787 (17)	0.1009 (19)	−0.0156 (12)	−0.0066 (12)	0.0080 (15)
N21	0.0467 (8)	0.0246 (7)	0.0441 (8)	0.0005 (6)	0.0060 (7)	0.0044 (6)
C27	0.0407 (9)	0.0282 (8)	0.0453 (9)	−0.0007 (7)	−0.0055 (7)	−0.0015 (7)
O27	0.0710 (9)	0.0261 (7)	0.0799 (10)	0.0005 (6)	0.0096 (8)	0.0023 (6)
C21	0.0409 (9)	0.0403 (9)	0.0416 (9)	0.0053 (8)	−0.0013 (7)	−0.0015 (8)
C22	0.0408 (9)	0.0486 (11)	0.0484 (10)	−0.0030 (8)	0.0033 (8)	0.0005 (9)
C23	0.0450 (11)	0.0761 (16)	0.0668 (14)	−0.0076 (11)	0.0052 (10)	0.0154 (12)
C24	0.0461 (11)	0.115 (2)	0.0524 (13)	0.0162 (14)	0.0085 (10)	0.0165 (14)
C25	0.0756 (16)	0.098 (2)	0.0515 (13)	0.0335 (16)	0.0116 (12)	−0.0083 (13)
C26	0.0698 (14)	0.0551 (13)	0.0577 (12)	0.0145 (11)	0.0051 (11)	−0.0102 (11)
S31	0.0482 (3)	0.0679 (4)	0.0544 (3)	−0.0136 (3)	−0.0010 (2)	0.0058 (3)
C32	0.0417 (9)	0.0525 (11)	0.0464 (10)	0.0021 (9)	0.0005 (8)	0.0004 (9)
C33	0.0534 (12)	0.0997 (19)	0.0464 (11)	−0.0023 (13)	0.0012 (9)	−0.0019 (12)
C34	0.0644 (14)	0.0890 (18)	0.0547 (13)	−0.0059 (13)	−0.0158 (11)	−0.0020 (12)
C35	0.0551 (12)	0.0722 (15)	0.0697 (14)	−0.0126 (12)	−0.0142 (11)	0.0088 (12)

Geometric parameters (Å, º)

C1—O1	1.204 (2)	C22—C23	1.389 (3)
C1—O2	1.336 (2)	C22—H22	0.9300
C1—C2	1.479 (3)	C23—C24	1.371 (4)
C2—C3	1.336 (2)	C23—H23	0.9300
C2—N21	1.417 (2)	C24—C25	1.368 (4)
C3—C32	1.439 (3)	C24—H24	0.9300
C3—H3	0.9300	C25—C26	1.381 (3)
O2—C4	1.445 (2)	C25—H25	0.9300
C4—H4A	0.9600	C26—H26	0.9300

C4—H4B	0.9600	S31—C35	1.701 (2)
C4—H4C	0.9600	S31—C32	1.7214 (19)
N21—C27	1.345 (2)	C32—C33	1.369 (3)
N21—H21	0.83 (2)	C33—C34	1.401 (3)
C27—O27	1.225 (2)	C33—H33	0.9300
C27—C21	1.493 (3)	C34—C35	1.341 (3)
C21—C22	1.383 (3)	C34—H34	0.9300
C21—C26	1.384 (3)	C35—H35	0.9300

O1—C1—O2	123.42 (18)	C23—C22—H22	120.0
O1—C1—C2	123.96 (18)	C24—C23—C22	120.3 (2)
O2—C1—C2	112.61 (16)	C24—C23—H23	119.8
C3—C2—N21	121.70 (16)	C22—C23—H23	119.8
C3—C2—C1	123.07 (16)	C25—C24—C23	120.0 (2)
N21—C2—C1	115.21 (15)	C25—C24—H24	120.0
C2—C3—C32	129.06 (17)	C23—C24—H24	120.0
C2—C3—H3	115.5	C24—C25—C26	120.2 (2)
C32—C3—H3	115.5	C24—C25—H25	119.9
C1—O2—C4	116.16 (17)	C26—C25—H25	119.9
O2—C4—H4A	109.5	C25—C26—C21	120.5 (2)
O2—C4—H4B	109.5	C25—C26—H26	119.8
H4A—C4—H4B	109.5	C21—C26—H26	119.8
O2—C4—H4C	109.5	C35—S31—C32	91.79 (11)
H4A—C4—H4C	109.5	C33—C32—C3	124.63 (18)
H4B—C4—H4C	109.5	C33—C32—S31	109.75 (15)
C27—N21—C2	121.66 (15)	C3—C32—S31	125.59 (14)
C27—N21—H21	118.2 (14)	C32—C33—C34	113.8 (2)
C2—N21—H21	119.4 (14)	C32—C33—H33	123.1
O27—C27—N21	121.27 (17)	C34—C33—H33	123.1
O27—C27—C21	121.26 (17)	C35—C34—C33	112.1 (2)
N21—C27—C21	117.46 (15)	C35—C34—H34	123.9
C22—C21—C26	119.06 (18)	C33—C34—H34	123.9
C22—C21—C27	122.81 (16)	C34—C35—S31	112.54 (18)
C26—C21—C27	118.12 (18)	C34—C35—H35	123.7
C21—C22—C23	119.9 (2)	S31—C35—H35	123.7
C21—C22—H22	120.0		

O1—C1—C2—C3	−179.81 (19)	C27—C21—C22—C23	178.87 (17)
O2—C1—C2—C3	−0.9 (3)	C21—C22—C23—C24	−0.9 (3)
O1—C1—C2—N21	−1.0 (3)	C22—C23—C24—C25	1.0 (3)
O2—C1—C2—N21	177.93 (15)	C23—C24—C25—C26	−0.2 (4)
N21—C2—C3—C32	−0.7 (3)	C24—C25—C26—C21	−0.9 (4)
C1—C2—C3—C32	178.00 (19)	C22—C21—C26—C25	1.1 (3)
O1—C1—O2—C4	−3.4 (3)	C27—C21—C26—C25	−178.04 (19)
C2—C1—O2—C4	177.66 (17)	C2—C3—C32—C33	−177.8 (2)
C3—C2—N21—C27	−101.8 (2)	C2—C3—C32—S31	−0.1 (3)
C1—C2—N21—C27	79.4 (2)	C35—S31—C32—C33	−0.28 (19)
C2—N21—C27—O27	3.9 (3)	C35—S31—C32—C3	−178.27 (19)

C2—N21—C27—C21	−176.77 (15)	C3—C32—C33—C34	178.3 (2)
O27—C27—C21—C22	161.07 (18)	S31—C32—C33—C34	0.3 (3)
N21—C27—C21—C22	−18.2 (3)	C32—C33—C34—C35	−0.2 (3)
O27—C27—C21—C26	−19.8 (3)	C33—C34—C35—S31	0.0 (3)
N21—C27—C21—C26	160.87 (17)	C32—S31—C35—C34	0.2 (2)
C26—C21—C22—C23	−0.2 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21—H21···O27^{i}	0.85 (2)	2.01 (2)	2.8038 (19)	158.1 (19)
C33—H33···*Cg*2^{ii}	0.93	2.68	3.472 (3)	144
C35—H35···*Cg*2^{iii}	0.93	2.92	3.684 (2)	140

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