Acta Crystallographica Section C
Structural Chemistry
ISSN 2053-2296

Response to *Heterocyclic tautomerism: reassignment of two crystal structures of 2-amino-1,3-thiazolidin-4-one derivatives* by Gzella *et al.* (2014)

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Received 8 July 2014
Accepted 21 July 2014

Prototropy tautomerism and *E*/*Z*-stereoisomerism of 2-iminothiazolidin-4-ones was extensively studied in the literature during the past several decades. This tautomerism was studied for both alkyl/aryl–amino/imino-substituted thiazolidinones/thiazolinones by several authors (Comrie, 1964; Åkerblom, 1967; Chowdry *et al.*, 2000; Mornon & Raveau, 1971; Ramsh *et al.*, 1978) in the solid state, as well as in solution, and the results can be generalized as follows. *Exo*-*N*-unsubstituted and *N*-alkyl-substituted compounds exist in solution preferentially as 2-(alkyl)aminothiazolin-4-ones, whereas *N*-aryl-substituted compounds prefer the 2-(arylimino)thiazolidin-4-one arrangement, although some exceptions to this rule were also reported (Sedlák *et al.*, 2002; Kammel & Hanusek, 2014). For example, in a recent article (Kammel & Hanusek, 2014) we have found that base-catalyzed cyclization of *N*-isopropyl-*S*-(2-oxo-2,3-dihydro-1-benzofuran-3-yl)isothiouronium bromide gives 5-(2-hydroxyphenyl)-2-isopropylimino-1,3-thiazolidin-4-one, which during crystallization or simple heating undergoes the above-mentioned prototropy tautomerism to give 5-(2-hydroxyphenyl)-2-isopropylamino-1,3-thiazolin-4-one. From this observation it is evident that the solvent, purification method, temperature and other parameters can influence the position of tautomeric equilibrium and also the crystal packing. In our article from 2009 (Váňa *et al.*, 2009), the single crystal of 5-(2-hydroxyethyl)-2-[(pyridin-2-yl)imino]-1,3-thiazolidin-4-one was grown from dimethyl sulfoxide (DMSO), whereas the single crystal studied by Gzella *et al.* (2014) was grown from methanol from a sample previously prepared in refluxing ethanol. In order to support our refinement (Váňa *et al.*, 2009), the following arguments are given: three peaks assignable to the 'amino or imine' H atoms were located on our Fourier difference map under the given measurement conditions (150 K). Both tautomers were refined but based on the known NMR assignment, literature data and higher electron density we decided to refine the structure as the imine tautomer.

To conclude, we are surprised how Gzella *et al.* (2014) could suggest that our structure had an incorrectly-positioned H atom based on the measurement under different conditions on crystals grown from a different solvent, and last, but not least, from a disordered structure, when tautomerism is always a chemical equilibrium.

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 doi:10.1107/S2053229614016799

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Heterocyclic tautomerism: reassignment of two crystal structures of 2-amino-1,3-thiazolidin-4-one derivatives

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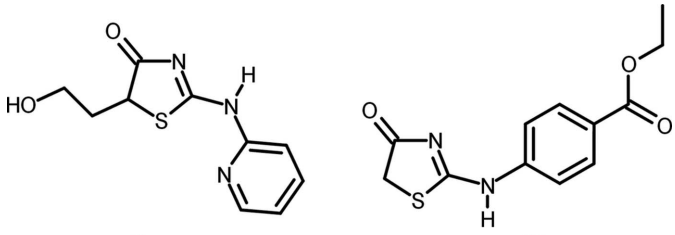
Received 8 May 2014
Accepted 27 June 2014

The structures of 5-(2-hydroxyethyl)-2-[(pyridin-2-yl)amino]-1,3-thiazolidin-4-one, $C_{10}H_{11}N_3O_2S$, (I), and ethyl 4-[(4-oxo-1,3-thiazolidin-2-yl)amino]benzoate, $C_{12}H_{12}N_2O_3S$, (II), which are identical to the entries with refcodes GACXOZ [Váňa *et al.* (2009). *J. Heterocycl. Chem.* **46**, 635–639] and HEGLUC [Behbehani & Ibrahim (2012). *Molecules*, **17**, 6362–6385], respectively, in the Cambridge Structural Database [Allen (2002). *Acta Cryst.* B**58**, 380–388], have been redetermined at 130 K. This structural study shows that both investigated compounds exist in their crystal structures as the tautomer with the carbonyl–imine group in the five-membered heterocyclic ring and an exocyclic amine N atom, rather than the previously reported tautomer with a secondary amide group and an exocyclic imine N atom. The physicochemical and spectroscopic data of the two investigated compounds are the same as those of GACXOZ and HEGLUC, respectively. In the thiazolidin-4-one system of (I), the S and chiral C atoms, along with the hydroxyethyl group, are disordered. The thiazolidin-4-one fragment takes up two alternative locations in the crystal structure, which allows the molecule to adopt *R* and *S* configurations. The occupancy factors of the disordered atoms are 0.883 (2) (for the *R* configuration) and 0.117 (2) (for the *S* configuration). In (I), the main factor that determines the crystal packing is a system of hydrogen bonds, involving both strong N—H···N and O—H···O and weak C—H···O hydrogen bonds, linking the molecules into a three-dimensional hydrogen-bond network. On the other hand, in (II), the molecules are linked *via* N—H···O hydrogen bonds into chains.

Keywords: crystal structure; amine–imine tautomerism; hydrogen bonding; 2-amino(imino)-1,3-thiazolidin-4-ones; pharmaceutical compounds; heterocyclic tautomerism; medicinal chemistry; biologically active compounds.

1. Introduction

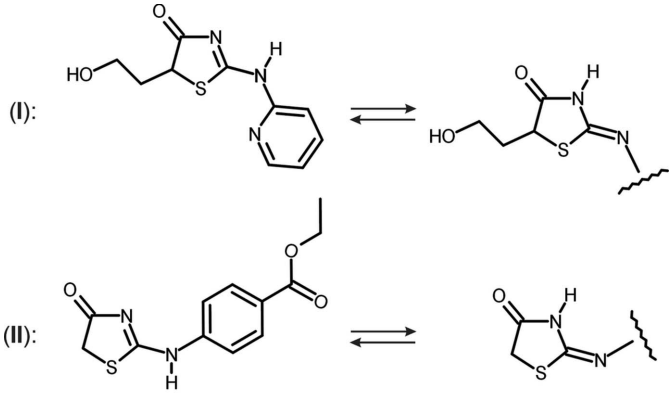
1,3-Thiazolidin-4-one derivatives are a well known class of patented drugs, examples being hypoglycaemic thiazolidinediones (pioglitazone and its analogues), aldose reductase inhibitors (epalrestat), anti-inflammatory agents (darbufelone) and new-generation diuretics (etozoline). In modern medicinal chemistry, the thiazolidinone core is a powerful biophore for the rational design of 'drug-like' molecules. Modern research into the pharmacological potential of 1,3-thiazolidin-4-ones has allowed the establishment of a wide spectrum of pharmacological activities, including anticancer, anti-inflammatory, antiviral, antiparasitic, antimicrobial and anti-oxidant (Lesyk & Zimenkovsky, 2004; Lesyk *et al.*, 2011).



(I) (II)

Scheme 1

It is worth noting that studies regarding amino–imino tautomerism in 2-amino(imino)-1,3-thiazolidin-4-one derivatives have been ongoing for almost 50 years. The investigations have been performed on crystalline and liquid phases using different spectroscopic techniques, *e.g.* IR, UV, and ^{1}H and ^{13}C NMR, or sometimes with quantum-chemical calculations. X-ray crystallography is not commonly used among the analytical methods applied to structural studies concerning



*A*1 (amine form) *A*2 (imine form)

Scheme 2

tautomerism. In the Cambridge Structural Database (CSD, Version 5.35; Allen, 2002), we found only 21 structures with the amine form (refcodes EKELEL FIVPIJ, FOWQOY, IHUFAS, IMPTHA01, IMPTHA12, IMTAZO01, INMTZO, JOBGOW, KUKZUM, PACPIU, PATAZO, PTHAZO10, SALYOT, SINQOW, SINQUC, TEBDAH, ULACAM, VELBEU, WOSMAS and YUQCAP) and 16 with the imine form (refcodes EHITZO, GACXOZ, HEGLUC, HEGMAJ,

  doi:10.1107/S2053229614015162

Table 1
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{10}H_{11}N_3O_2S$	$C_{12}H_{12}N_2O_3S$
M_r	237.28	264.30
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
Temperature (K)	130	130
a, b, c (Å)	5.78910 (15), 8.8045 (2), 10.9688 (3)	3.9850 (2), 5.5113 (3), 26.8877 (14)
α, β, γ (°)	90.638 (2), 95.794 (2), 107.990 (2)	84.483 (5), 89.670 (5), 86.338 (5)
V (Å^3)	528.50 (3)	586.58 (6)
Z	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.29	0.28
Crystal size (mm)	0.42 × 0.22 × 0.10	0.40 × 0.34 × 0.05
Data collection		
Diffractometer	Agilent Xcalibur Atlas diffractometer	Agilent Xcalibur Atlas diffractometer
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2012)	Multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min}, T_{max}	0.939, 1.000	0.908, 1.000
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	5332, 2501, 2338	7855, 2804, 2604
R_{int}	0.016	0.017
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.684	0.684
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.088, 1.13	0.030, 0.078, 1.05
No. of reflections	2501	2804
No. of parameters	167	168
No. of restraints	4	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.21	0.54, −0.23

Computer programs: *CrysAlis PRO* (Agilent, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012), *WinGX* (Farrugia, 2012), *OLEX2* (Dolomanov *et al.*, 2009) and *PLATON* (Spek, 2009).

HEGMEN, HEGMIR, HEGMOX, IMTAZO, IOTAGP, IXTAZD10, RIPMOT, ROMXUN, SOHHIH, ULACEQ, VAMPUW and YARLIN), the latter set including two structures, GACXOZ (Váňa *et al.*, 2009) and HEGLUC (Behbehani & Ibrahim, 2012), with an incorrectly specified tautomeric form, as will be demonstrated here.

As part of a programme aimed at the development of new biologically active compounds, we have prepared 5-(2-hydroxyethyl)-2-[(pyridin-2-yl)amino]-1,3-thiazolidin-4-one, (I) (*cf.* CSD refcode GACXOZ), and ethyl 4-[(4-oxo-1,3-thiazolidin-2-yl)amino]benzoate, (II) (*cf.* CSD refcode HEGLUC) (Scheme 1), and have made corrections to the interpretations of their previously published structures. According to the previous reports, the investigated compounds exist in tautomeric form *A*2, with a secondary amide group in the five-membered heterocyclic ring and an exocyclic imine N atom (see Scheme 2).

2. Experimental

2.1. Synthesis and crystallization

The title compounds were synthesized by methods used for obtaining 2-amino(imino)-1,3-thiazolidin-4-one derivatives (Subtel'na *et al.*, 2010; Geronikaki *et al.*, 2008; Ostapiuk *et al.*, 2012). Compound (I) was prepared by the [2+3]-cyclocondensation reaction of 3-bromotetrahydrofuran-2-one (α-bromo-γ-butyrolactone) with 1-(pyridin-2-yl)thiourea in the presence of fused sodium acetate in refluxing ethanol. Compound (II) was synthesized through cyclocondensation of ethyl 4-(2-chloroacetylamino)benzoate and ammonium thiocyanate in ethanol (Behbehani & Ibrahim, 2012). It is known that the above-mentioned reactions do not stop at the nucleophilic substitution stage (Geronikaki *et al.*, 2008; Ostapiuk *et al.*, 2012). The intermediate α-thiocyanatoamide undergoes spontaneous cyclization/rearrangement to give the thiazolidin-4-one derivative, (II).

The physicochemical and spectroscopic data of (I) and (II) (see *Supporting information*) are the same as for GACXOZ and HEGLUC, respectively. Crystals suitable for single-crystal X-ray diffraction analysis were grown by slow evaporation of solutions in methanol [for (I)] and dimethylformamide [for (II)].

2.2. Refinement

For both (I) and (II), N-bound H atoms were obtained from difference Fourier maps and refined freely and isotropically. The remaining H atoms were positioned geometrically and refined within the riding-model approximation, with methyl C—H = 0.98 Å, methylene C—H = 0.99 Å, methine C—H = 1.00 Å, Csp^2 C—H = 0.95 Å and O—H = 0.88 Å, and with U_{iso}(H) = 1.5U_{eq}(C,O) for methyl and hydroxy H atoms, or 1.2U_{eq}(C) otherwise. The methyl groups were refined as rigid groups, which were allowed to rotate. Non-H atoms of the disordered part of the molecule of (I) were obtained from

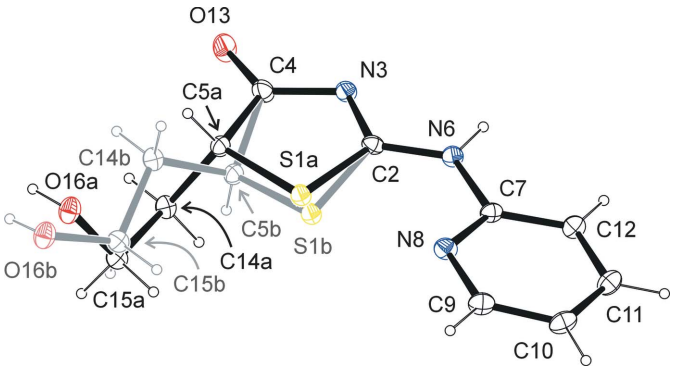


Figure 1
The molecular structure of (I), showing the atomic labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. The disordered part *b* of the molecule is coloured grey, as distinct from the major component shown in black.

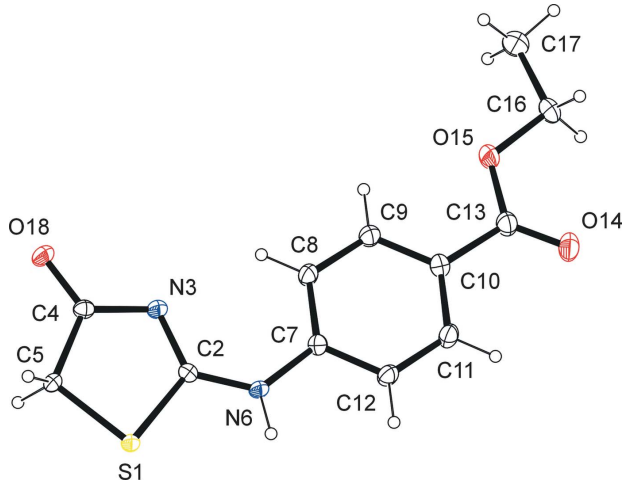


Figure 2
A view of the molecule of (II), showing the atomic labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

difference Fourier maps. During refinement, the atomic displacement ellipsoids of the corresponding atoms in the alternative conformations, *a* and *b*, were constrained to be identical. Corresponding bond distances within the two disordered components were restrained to be similar. The H atoms of the –OH groups of the hydroxyethyl residues in positions *a* and *b* were separated in an arbitrary manner by applying a H···H distance restraint of 0.61 (2) Å.

3. Results and discussion

3.1. Tautomeric forms

Our revision of the X-ray studies has shown that, in their crystal structures, compounds (I) and (II) adopt tautomeric form *A*1 rather than the previously suggested form *A*2 (Scheme 2, and Figs. 1 and 2). In both structures, the H atom was located at the exocyclic N atom (N6). This observation for (II) is supported by the presence of an intermolecular N6—H6···O18^{i} hydrogen bond (Table 2 and Fig. 3), in which atom N6 acts as a proton donor and carbonyl atom O18 acts as a

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H6···O18^{i}	0.857 (17)	1.954 (17)	2.7882 (14)	164.3 (17)

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

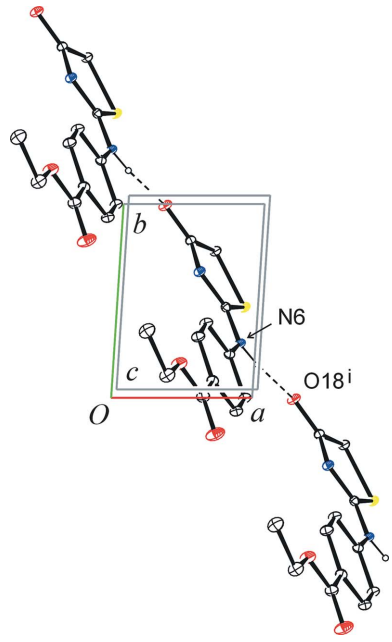


Figure 3
Part of the crystal structure of (II), showing the formation of the hydrogen-bonded chain along [110]. See Table 2 for symmetry code. H atoms not involved in hydrogen bonding (dashed lines) have been omitted for clarity.

proton acceptor. The formation of this N—H···O hydrogen bond is promoted by the antiperiplanar conformation of the C2—N3 and N6—H6 bonds [torsion angle N3—C2—N6—H6 = 180 (2)°].

In (I), the presence of an intermolecular N6—H6···N3^{i} hydrogen bond between the amidine groups (Table 3 and Fig. 4*a*) may lead to ambiguity about the amine/imine character of atoms N3 and N6. It is known that the presence of N—H···N hydrogen-bond contacts enhances the resonance effect, which is significant even for unassociated molecules. Therefore, one may be inclined to think that, for structure (I) forming hydrogen-bonded dimers, both tautomeric forms are possible, and that the C2—N3 and C2—N6 bond lengths do

Table 3
Hydrogen-bond geometry (Å, °) for (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H6···N3^{i}	0.88 (2)	2.10 (2)	2.9733 (17)	173 (2)
O16*a*—H16*A*···O13^{ii}	0.84	2.00	2.7940 (19)	158
O16*b*—H16*B*···O13^{ii}	0.84	1.86	2.692 (12)	168
C10—H10···O16*a*^{iii}	0.95	2.52	3.442 (2)	164
C12—H12···O13^{i}	0.95	2.31	3.199 (2)	155
C14*a*—H14*B*···O16*a*^{iv}	0.99	2.54	3.448 (2)	152

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

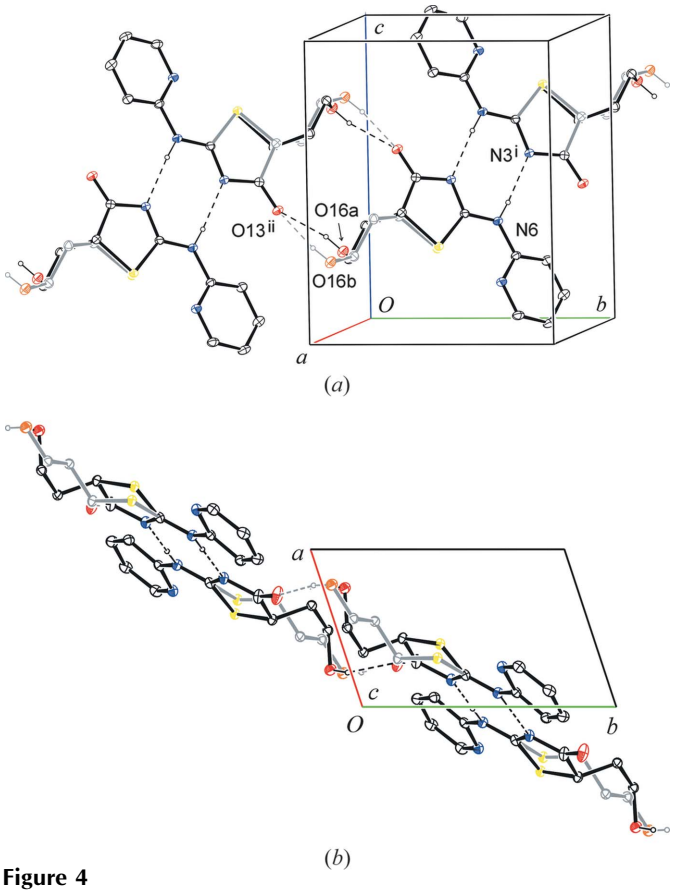


Figure 4
(*a*) The molecular tape in (I), generated by the centrosymmetric dimers. See Table 3 for symmetry codes. H atoms not involved in hydrogen bonding (dashed lines) have been omitted for clarity. (*b*) The molecular tape in (I), expanded along the [110] direction. Grey shading denotes the disordered part of the molecule.

not provide much useful information for solving this problem because of resonance interactions which render their lengths similar regardless of the tautomeric form. However, atom H6 was located and refined at this position and, in addition, analysis of the C2—N3 and C2—N6 bond lengths performed for tautomeric forms *A*1 and *A*2 did not confirm these suppositions.

The average C2—N3 and C2—N6 bond lengths in 20 2-amino-1,3-thiazolidin-4-one derivatives deposited in the CSD and exhibiting the *A*1 tautomeric form are similar and have values of 1.325 (1) and 1.315 (2) Å, respectively (refcodes EKELEL, FIVPIJ, FOBQOY, IHUFAS, IMPTHA12, IMTAZO01, INMTZO, JOBGOW, KUKZUM, PACPIU, PTHAZO10, SALYOT, SINQOW, SINQUC, TEBDAH, ULACAM, VELBEU, VEQFAA, WOSMAS and YUQCAP; $R < 0.07$). These mean C2—N3 and C2—N6 bond lengths are intermediate between the lengths of single and double C—N bonds. In comparison with the normal literature C═N double-bond value of 1.279 (1) Å (Allen *et al.*, 1987), they are lengthened by about 33σ and 16σ, respectively. On the other hand, they are shortened by about 26σ and 24σ, respectively, compared with the mean value for a Csp^2—N single-bond length of 1.383 (2) Å. This latter value was obtained from 117 structures of 2-imino-1,3-thiazolidin-4-one derivatives substituted at N3 ($R < 0.07$). Based on six records revealing the *A*2 tautomeric form, the average C2—N3 and C2—N6 bond lengths were calculated as 1.374 (3) and 1.280 (2) Å, respectively (refcodes EHITZO, HEGMAJ, HEGMEN, HEGMIR, HEGMOX and VAMPUV; $R < 0.07$), which are clearly different from one another. The former is similar to the normal Csp^2—N single-bond length in heterocyclic rings, while the latter is close to a normal C═N double-bond length.

Our observations thus indicate an unequal resonance effect in tautomeric forms *A*1 and *A*2, which allows the use of the C—N bond lengths to distinguish the two forms. Crystal structure analysis of (I) and (II) shows that the interatomic lengths C2—N3 and C2—N6 [1.3256 (17) and 1.3385 (19) Å in (I), and 1.3182 (15) and 1.3282 (15) Å in (II), respectively] have comparable values, which is a typical feature of tautomeric form *A*1.

We thus submit that the original tautomeric assignments of GACXOZ and HEGLUC were incorrect, and propose they both be reassigned to the *A*1 form, supported by the evidence presented here. From the comparison of (I)/GACXOZ and (II)/HEGLUC it is clear that the molecules in pairs have the same geometry and, what is particularly important, very similar C2—N3 and C2—N6 bond lengths. In the first pair, the bond lengths are 1.326/1.322 and 1.338/1.337 Å, while in the second pair the values are 1.318/1.308 and 1.328/1.333 Å, respectively. The structural differences relate to the position of the mobile N—H hydrogen only. We think that incorrect localization of the H atom at the endocyclic and not at the exocyclic N atom in GACXOZ and HEGLUC is most likely a result of the fact that the H atoms bonded to N atoms were positioned geometrically and were treated using a riding model, with the U_{iso}(H) parameter calculated and not refined. If the U_{iso}(H) parameter had been refined in the earlier report, the mistake would have been noticed and corrected.

3.2. Further details of the structural analysis

Some atoms in the crystal structure of (I) are disordered. This observation concerns the part of the molecule that includes atoms S1, C5, C14, C15 and O16 of the 2-hydroxyethyl-1,3-thiazolidin-4-one fragment. Each of these atoms takes up two alternative locations in the crystal structure, labelled *a* and *b*. This arrangement results in two different enantiomers of the molecule, with atoms S1, C5, C14, C15 and O16 in position *a* having an *R* configuration and the atoms in position *b* having an *S* configuration (for the molecule shown in Fig. 1), and *vice versa* for a symmetry-related site. The occupancy factor for these five atoms in orientation *a* is 0.883 (2), while in orientation *b* the occupancy factor is 0.117 (2).

The thiazolidine ring with atoms S1 and C5 in arrangement *a* is approximately planar (r.m.s. deviation = 0.0213 Å), while the ring with these atoms in arrangement *b* is folded (r.m.s. deviation = 0.1130 Å) and adopts a half-chair conformation [Cremer & Pople (1975) puckering parameters Q = 0.253 (8) Å and φ = 130.0 (16)°].

The pairs of bonds S1—C2/N6—C7 and C2—N6/C7—N8 are in a synperiplanar conformation. The torsion angles S1*a*/S1*b*—C2—N6—C7 and C2—N6—C7—N8 are 4.2 (2)/−14.7 (3) and −10.6 (2)°, respectively. The arrangements of the two alternative hydroxyethyl residues are determined by torsion angles C5*a*—14*a*—C15*a*—O16*a* = 55.8 (2)° and C5*b*—C14*b*—C15*b*—O16*b* = 178.1 (14)°, from which the pairs of bonds C5*a*—C14*a*/C15*a*—O16*a* and C5*b*—C14*b*/C15*b*—O16*b* are synclinal and synperiplanar, respectively.

The heterocyclic and benzene rings in (II) are both flat and approximately coplanar. The dihedral angle between their mean planes is 6.59 (6)°. Atoms C13, O14, O15 and C16 form a flat system (r.m.s. deviation = 0.0015 Å) that is twisted out of the mean plane of the benzene ring by 2.23 (8)°. The remaining atom C17 is tilted from the planar system formed by atoms C13, O14, O15 and C16 and is 0.201 (3) Å from that plane. Atoms C13 and C17 are in an antiperiplanar conformation [torsion angle C13—O15—C16—C17 = 172.17 (12)°]. On the other hand, the C13═O14 carbonyl group is in a synperiplanar conformation relative to the S1—C2 bond of the heterocyclic ring [torsion angle S1—C2···C13—O14 = −1.83 (18)°].

The partial double-bond character of the C2—N6 bond in (I) and (II) accounts for the hindered rotation of the pyridin-2-ylamino [in (I)] or phenylamino [in (II)] residues in the analysed structures. The dihedral angles between the cyclic systems are 11.67 (11)/27.4 (4)° for (I) and 6.59 (6)° for (II). The two values given for (I) result from the previously described disorder concerning the arrangement of atoms S1 and C5 in the crystal structure.

The main factor that determines the crystal packing and the formation of the supramolecular structure of (I) is the system of hydrogen bonds, involving both strong N—H···N and O—H···O and weak C—H···O hydrogen bonds (Table 3). The N6—H6···N3^{i} and C12—H12···O13^{i} hydrogen bonds link (symmetry codes as in Table 3) the molecules into centrosymmetric dimers and generate centrosymmetric $R_2^2(8)$ (Bernstein *et al.*, 1995) ring motifs. Neighbouring dimers are linked through O16*a*—H16*A*···O13^{ii} and O16*b*—H16*B*···O13^{ii} hydrogen bonds to form the next centrosymmetric ring motif of $R_2^2(14)$ type (Fig. 4*a*). These contacts link the molecules of (I) into tapes extending along the [110] direction (Table 3, and Figs. 4*a* and 4*b*). Neighbouring molecular tapes are linked through nonclassical C10—H10···O16^{iii} hydrogen bonds into layers parallel to the (111) plane, and these layers are connected by C14*a*—H14*B*···O16*a*^{iv} hydrogen bonds, forming a three-dimensional hydrogen-bond network.

The molecules of (II) are linked in the crystal structure through N6—H6···O18^{i} hydrogen bonds into chains extending along the [110] direction (Table 2 and Fig. 3).

Supporting information for this paper is available from the IUCr electronic archives (Reference: WQ3065).

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Acta Cryst. (2014). C**70**, 812-816 [doi:10.1107/S2053229614015162]

Heterocyclic tautomerism: reassignment of two crystal structures of 2-amino-1,3-thiazolidin-4-one derivatives

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Computing details

For both compounds, data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis PRO* (Agilent, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012), OLEX2 (Dolomanov *et al.*, 2009) and *PLATON* (Spek, 2009).

(I) 5-(2-Hydroxyethyl)-2-[(pyridin-2-yl)amino]-1,3-thiazolidin-4-one

Crystal data

$C_{10}H_{11}N_3O_2S$
$M_r = 237.28$
Triclinic, $P\overline{1}$
$a = 5.78910\ (15)$ Å
$b = 8.8045\ (2)$ Å
$c = 10.9688\ (3)$ Å
$\alpha = 90.638\ (2)°$
$\beta = 95.794\ (2)°$
$\gamma = 107.990\ (2)°$
$V = 528.50\ (3)$ Å^3
$Z = 2$
$F(000) = 248$
$D_x = 1.491$ Mg m^{-3}
Melting point = 459–461 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3632 reflections
$\theta = 3.0–29.1°$
$\mu = 0.29$ mm^{-1}
$T = 130$ K
Lath, colourless
0.42 × 0.22 × 0.10 mm

Data collection

Agilent Xcalibur Atlas diffractometer
Radiation source: Enhance (Mo) X-ray Source
Detector resolution: 10.3088 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
$T_{min} = 0.939$, $T_{max} = 1.000$
5332 measured reflections
2501 independent reflections
2338 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 29.1°$, $\theta_{min} = 2.4°$
$h = -6\rightarrow7$
$k = -11\rightarrow11$
$l = -14\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.088$
$S = 1.13$
2501 reflections
167 parameters
4 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0328P)^2 + 0.2306P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.34$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1A	0.40161 (15)	0.38579 (7)	0.27170 (4)	0.01982 (15)	0.883 (2)
C2	0.2062 (3)	0.44461 (16)	0.36246 (12)	0.0199 (3)	
N3	0.1772 (2)	0.38288 (14)	0.47166 (10)	0.0214 (3)	
C4	0.2935 (3)	0.26908 (18)	0.48923 (13)	0.0267 (3)	
N6	0.0916 (2)	0.55040 (14)	0.32764 (11)	0.0206 (3)	
H6	0.004 (4)	0.572 (2)	0.3822 (18)	0.034 (5)*	
C7	0.0942 (3)	0.62302 (16)	0.21345 (12)	0.0202 (3)	
N8	0.2530 (2)	0.60269 (15)	0.14094 (11)	0.0230 (3)	
C9	0.2538 (3)	0.66848 (18)	0.03061 (13)	0.0259 (3)	
H9	0.3640	0.6533	−0.0234	0.031*	
C10	0.1021 (3)	0.75713 (19)	−0.00803 (13)	0.0274 (3)	
H10	0.1087	0.8026	−0.0862	0.033*	
C11	−0.0599 (3)	0.77766 (19)	0.07079 (14)	0.0274 (3)	
H11	−0.1652	0.8388	0.0472	0.033*	
C12	−0.0682 (3)	0.70917 (18)	0.18365 (13)	0.0233 (3)	
H12	−0.1794	0.7202	0.2386	0.028*	
O13	0.2882 (3)	0.19347 (15)	0.58229 (10)	0.0383 (3)	
C15A	0.5049 (3)	0.0412 (2)	0.2396 (2)	0.0218 (4)	0.883 (2)
H15A	0.4441	−0.0734	0.2128	0.026*	0.883 (2)
H15B	0.4880	0.1044	0.1670	0.026*	0.883 (2)
C5A	0.4400 (3)	0.2454 (2)	0.38628 (14)	0.0206 (3)	0.883 (2)
H5A	0.6166	0.2736	0.4186	0.025*	0.883 (2)
C14A	0.3496 (3)	0.07226 (19)	0.33477 (15)	0.0219 (3)	0.883 (2)
H14A	0.3511	−0.0003	0.4030	0.026*	0.883 (2)
H14B	0.1786	0.0475	0.2970	0.026*	0.883 (2)
O16A	0.7567 (2)	0.08204 (17)	0.28591 (13)	0.0269 (3)	0.883 (2)
H16A	0.7756	0.0159	0.3375	0.040*	0.883 (2)
S1B	0.3118 (10)	0.3571 (5)	0.2556 (4)	0.01982 (15)	0.117 (2)
C5B	0.312 (3)	0.2013 (16)	0.3623 (11)	0.0206 (3)	0.117 (2)
H5B	0.1685	0.1039	0.3387	0.025*	0.117 (2)
C14B	0.547 (2)	0.1577 (14)	0.3752 (11)	0.0219 (3)	0.117 (2)
H14C	0.5457	0.0840	0.4428	0.026*	0.117 (2)
H14D	0.6892	0.2551	0.3939	0.026*	0.117 (2)
C15B	0.564 (3)	0.077 (2)	0.2534 (17)	0.0218 (4)	0.117 (2)
H15C	0.4218	−0.0207	0.2370	0.026*	0.117 (2)
H15D	0.5559	0.1502	0.1864	0.026*	0.117 (2)

O16B	0.778 (2)	0.0357 (15)	0.2525 (11)	0.0269 (3)	0.117 (2)
H16B	0.7745	−0.0389	0.3001	0.040*	0.117 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1A	0.0243 (3)	0.0197 (2)	0.0173 (2)	0.0081 (2)	0.0067 (2)	0.00383 (16)
C2	0.0251 (7)	0.0173 (6)	0.0162 (6)	0.0040 (5)	0.0048 (5)	0.0009 (5)
N3	0.0285 (7)	0.0208 (6)	0.0167 (5)	0.0093 (5)	0.0056 (5)	0.0035 (4)
C4	0.0405 (9)	0.0245 (7)	0.0194 (7)	0.0151 (7)	0.0078 (6)	0.0030 (6)
N6	0.0242 (6)	0.0225 (6)	0.0167 (5)	0.0082 (5)	0.0065 (5)	0.0046 (5)
C7	0.0233 (7)	0.0186 (6)	0.0163 (6)	0.0028 (5)	0.0025 (5)	0.0034 (5)
N8	0.0280 (7)	0.0226 (6)	0.0186 (6)	0.0067 (5)	0.0065 (5)	0.0041 (5)
C9	0.0286 (8)	0.0287 (8)	0.0189 (7)	0.0052 (6)	0.0073 (6)	0.0045 (6)
C10	0.0291 (8)	0.0307 (8)	0.0190 (7)	0.0039 (6)	0.0032 (6)	0.0095 (6)
C11	0.0259 (8)	0.0294 (8)	0.0256 (7)	0.0072 (6)	0.0011 (6)	0.0089 (6)
C12	0.0228 (7)	0.0247 (7)	0.0221 (7)	0.0061 (6)	0.0045 (5)	0.0045 (6)
O13	0.0691 (9)	0.0381 (7)	0.0218 (5)	0.0333 (6)	0.0163 (5)	0.0136 (5)
C15A	0.0223 (11)	0.0206 (11)	0.0222 (8)	0.0058 (8)	0.0040 (8)	0.0014 (7)
C5A	0.0244 (9)	0.0209 (8)	0.0184 (7)	0.0095 (7)	0.0032 (6)	0.0034 (6)
C14A	0.0216 (8)	0.0198 (8)	0.0248 (8)	0.0064 (6)	0.0052 (6)	0.0025 (6)
O16A	0.0254 (7)	0.0284 (8)	0.0295 (8)	0.0108 (5)	0.0069 (5)	0.0088 (6)
S1B	0.0243 (3)	0.0197 (2)	0.0173 (2)	0.0081 (2)	0.0067 (2)	0.00383 (16)
C5B	0.0244 (9)	0.0209 (8)	0.0184 (7)	0.0095 (7)	0.0032 (6)	0.0034 (6)
C14B	0.0216 (8)	0.0198 (8)	0.0248 (8)	0.0064 (6)	0.0052 (6)	0.0025 (6)
C15B	0.0223 (11)	0.0206 (11)	0.0222 (8)	0.0058 (8)	0.0040 (8)	0.0014 (7)
O16B	0.0254 (7)	0.0284 (8)	0.0295 (8)	0.0108 (5)	0.0069 (5)	0.0088 (6)

Geometric parameters (Å, º)

S1A—C2	1.7655 (15)	C12—H12	0.9500
S1A—C5A	1.8178 (16)	C15A—O16A	1.427 (2)
C2—N3	1.3256 (17)	C15A—C14A	1.520 (3)
C2—N6	1.3385 (19)	C15A—H15A	0.9900
C2—S1B	1.660 (5)	C15A—H15B	0.9900
N3—C4	1.3744 (19)	C5A—C14A	1.532 (2)
C4—O13	1.2227 (18)	C5A—H5A	1.0000
C4—C5A	1.528 (2)	C14A—H14A	0.9900
C4—C5B	1.535 (13)	C14A—H14B	0.9900
N6—C7	1.4119 (17)	O16A—H16A	0.8400
N6—H6	0.88 (2)	S1B—C5B	1.813 (13)
C7—N8	1.3251 (19)	C5B—C14B	1.520 (14)
C7—C12	1.396 (2)	C5B—H5B	1.0000
N8—C9	1.3473 (18)	C14B—C15B	1.533 (15)
C9—C10	1.384 (2)	C14B—H14C	0.9900
C9—H9	0.9500	C14B—H14D	0.9900
C10—C11	1.387 (2)	C15B—O16B	1.397 (16)
C10—H10	0.9500	C15B—H15C	0.9900

C11—C12	1.382 (2)	C15B—H15D	0.9900
C11—H11	0.9500	O16B—H16B	0.8400

C2—S1A—C5A	89.64 (7)	C14A—C15A—H15B	109.2
N3—C2—N6	118.87 (13)	H15A—C15A—H15B	107.9
N3—C2—S1B	120.45 (18)	C4—C5A—C14A	111.18 (14)
N6—C2—S1B	118.35 (18)	C4—C5A—S1A	105.24 (11)
N3—C2—S1A	117.77 (11)	C14A—C5A—S1A	112.27 (11)
N6—C2—S1A	123.35 (10)	C4—C5A—H5A	109.3
C2—N3—C4	111.24 (12)	C14A—C5A—H5A	109.3
O13—C4—N3	123.02 (14)	S1A—C5A—H5A	109.3
O13—C4—C5A	121.08 (14)	C15A—C14A—C5A	112.54 (14)
N3—C4—C5A	115.86 (12)	C15A—C14A—H14A	109.1
O13—C4—C5B	123.3 (5)	C5A—C14A—H14A	109.1
N3—C4—C5B	107.5 (4)	C15A—C14A—H14B	109.1
C2—N6—C7	126.46 (13)	C5A—C14A—H14B	109.1
C2—N6—H6	114.2 (13)	H14A—C14A—H14B	107.8
C7—N6—H6	119.3 (13)	C15A—O16A—H16A	109.5
N8—C7—C12	124.43 (13)	C2—S1B—C5B	87.4 (4)
N8—C7—N6	116.53 (12)	C14B—C5B—C4	105.5 (9)
C12—C7—N6	119.04 (13)	C14B—C5B—S1B	113.8 (9)
C7—N8—C9	117.15 (13)	C4—C5B—S1B	106.5 (7)
N8—C9—C10	123.26 (14)	C14B—C5B—H5B	110.3
N8—C9—H9	118.4	C4—C5B—H5B	110.3
C10—C9—H9	118.4	S1B—C5B—H5B	110.3
C9—C10—C11	118.06 (13)	C5B—C14B—C15B	107.7 (11)
C9—C10—H10	121.0	C5B—C14B—H14C	110.2
C11—C10—H10	121.0	C15B—C14B—H14C	110.2
C12—C11—C10	120.01 (14)	C5B—C14B—H14D	110.2
C12—C11—H11	120.0	C15B—C14B—H14D	110.2
C10—C11—H11	120.0	H14C—C14B—H14D	108.5
C11—C12—C7	117.06 (14)	O16B—C15B—C14B	113.1 (13)
C11—C12—H12	121.5	O16B—C15B—H15C	109.0
C7—C12—H12	121.5	C14B—C15B—H15C	109.0
O16A—C15A—C14A	112.19 (16)	O16B—C15B—H15D	109.0
O16A—C15A—H15A	109.2	C14B—C15B—H15D	109.0
C14A—C15A—H15A	109.2	H15C—C15B—H15D	107.8
O16A—C15A—H15B	109.2	C15B—O16B—H16B	109.5

C5A_a—S1A_a—C2—N3	4.74 (12)	C5B—C4—C5A—C14A	−43.3 (9)
C5A—S1A—C2—N6	−176.87 (13)	O13—C4—C5A—S1A	−178.17 (14)
C5A—S1A—C2—S1B	−99.0 (5)	N3—C4—C5A—S1A	−0.17 (18)
N6—C2—N3—C4	175.93 (13)	C5B—C4—C5A—S1A	78.4 (10)
S1B—C2—N3—C4	13.5 (3)	C2—S1A—C5A—C4	−2.25 (12)
S1A—C2—N3—C4	−5.60 (17)	C2—S1A—C5A—C14A	118.83 (13)
C2—N3—C4—O13	−178.53 (15)	O16A—C15A—C14A—C5A	55.8 (2)
C2—N3—C4—C5A	3.5 (2)	C4—C5A—C14A—C15A	−175.19 (15)
C2—N3—C4—C5B	−25.5 (6)	S1A—C5A—C14A—C15A	67.21 (17)

N3—C2—N6—C7	−177.41 (13)	N3—C2—S1B—C5B	3.1 (5)
S1B—C2—N6—C7	−14.7 (3)	N6—C2—S1B—C5B	−159.4 (5)
S1A—C2—N6—C7	4.2 (2)	S1A—C2—S1B—C5B	88.8 (7)
C2—N6—C7—N8	−10.6 (2)	O13—C4—C5B—C14B	−59.0 (11)
C2—N6—C7—C12	168.97 (14)	N3—C4—C5B—C14B	148.0 (7)
C12—C7—N8—C9	−0.9 (2)	C5A—C4—C5B—C14B	35.7 (7)
N6—C7—N8—C9	178.62 (12)	O13—C4—C5B—S1B	179.7 (3)
C7—N8—C9—C10	1.3 (2)	N3—C4—C5B—S1B	26.7 (8)
N8—C9—C10—C11	−0.6 (2)	C5A—C4—C5B—S1B	−85.6 (11)
C9—C10—C11—C12	−0.6 (2)	C2—S1B—C5B—C14B	−132.4 (10)
C10—C11—C12—C7	1.0 (2)	C2—S1B—C5B—C4	−16.6 (7)
N8—C7—C12—C11	−0.3 (2)	C4—C5B—C14B—C15B	175.6 (11)
N6—C7—C12—C11	−179.74 (13)	S1B—C5B—C14B—C15B	−67.9 (14)
O13—C4—C5A—C14A	60.0 (2)	C5B—C14B—C15B—O16B	178.1 (14)
N3—C4—C5A—C14A	−121.96 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H6···N3^{i}	0.88 (2)	2.10 (2)	2.9733 (17)	173 (2)
O16a—H16*A*···O13^{ii}	0.84	2.00	2.7940 (19)	158
O16b—H16*B*···O13^{ii}	0.84	1.86	2.692 (12)	168
C10—H10···O16a^{iii}	0.95	2.52	3.442 (2)	164
C12—H12···O13^{i}	0.95	2.31	3.199 (2)	155
C14a—H14*B*···O16a^{iv}	0.99	2.54	3.448 (2)	152

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

(II) Ethyl 4-[(4-oxothiazolidin-2-yl)amino]benzoate

Crystal data

$C_{12}H_{12}N_2O_3S$
M_r = 264.30
Triclinic, $P\overline{1}$
a = 3.9850 (2) Å
b = 5.5113 (3) Å
c = 26.8877 (14) Å
α = 84.483 (5)°
β = 89.670 (5)°
γ = 86.338 (5)°
V = 586.58 (6) Å^3
Z = 2
F(000) = 276
D_x = 1.496 Mg m^{-3}
Melting point = 461–462 K
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 5612 reflections
θ = 2.3–29.0°
μ = 0.28 mm^{-1}
T = 130 K
Lath, colourless
0.40 × 0.34 × 0.05 mm

Data collection

Agilent Xcalibur Atlas diffractometer
Radiation source: Enhance (Mo) X-ray Source
Detector resolution: 10.3088 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.908, T_{max} = 1.000
7855 measured reflections
2804 independent reflections
2604 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 29.1°, θ_{min} = 2.3°
h = −5→5
k = −7→7
l = −35→35

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.078$
$S = 1.05$
2804 reflections
168 parameters
0 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 0.3017P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ 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}
S1	0.90166 (7)	0.46485 (5)	0.05976 (2)	0.01454 (9)
C2	0.7690 (3)	0.4621 (2)	0.12301 (4)	0.0130 (2)
N3	0.5653 (3)	0.64554 (18)	0.13492 (4)	0.0152 (2)
C4	0.4898 (3)	0.8077 (2)	0.09439 (4)	0.0145 (2)
C5	0.6724 (3)	0.7546 (2)	0.04612 (4)	0.0151 (2)
H5A	0.8277	0.8836	0.0360	0.018*
H5B	0.5092	0.7468	0.0188	0.018*
N6	0.8822 (3)	0.27244 (18)	0.15391 (4)	0.0148 (2)
H6	1.014 (4)	0.167 (3)	0.1409 (7)	0.028 (4)*
C7	0.8121 (3)	0.2168 (2)	0.20533 (4)	0.0142 (2)
C8	0.6325 (3)	0.3760 (2)	0.23453 (5)	0.0177 (2)
H8	0.5475	0.5321	0.2203	0.021*
C9	0.5797 (3)	0.3034 (2)	0.28469 (5)	0.0188 (3)
H9	0.4585	0.4111	0.3049	0.023*
C10	0.7019 (3)	0.0748 (2)	0.30575 (4)	0.0168 (2)
C11	0.8843 (3)	−0.0814 (2)	0.27639 (5)	0.0185 (2)
H11	0.9711	−0.2368	0.2908	0.022*
C12	0.9398 (3)	−0.0119 (2)	0.22645 (5)	0.0174 (2)
H12	1.0643	−0.1191	0.2065	0.021*
C13	0.6394 (3)	−0.0095 (2)	0.35919 (5)	0.0198 (3)
O14	0.7419 (3)	−0.20429 (19)	0.37957 (4)	0.0322 (3)
O15	0.4537 (3)	0.15882 (17)	0.38186 (3)	0.0237 (2)
C16	0.3732 (4)	0.0971 (3)	0.43390 (5)	0.0244 (3)
H16A	0.5800	0.0440	0.4532	0.029*
H16B	0.2180	−0.0367	0.4374	0.029*
C17	0.2087 (4)	0.3256 (3)	0.45242 (5)	0.0288 (3)
H17A	0.0085	0.3785	0.4323	0.043*
H17B	0.3674	0.4549	0.4496	0.043*
H17C	0.1435	0.2917	0.4875	0.043*
O18	0.2933 (2)	0.98859 (16)	0.09484 (3)	0.01938 (19)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01742 (16)	0.01331 (14)	0.01236 (14)	0.00285 (11)	0.00282 (10)	−0.00122 (10)
C2	0.0132 (5)	0.0136 (5)	0.0126 (5)	−0.0021 (4)	0.0011 (4)	−0.0021 (4)
N3	0.0167 (5)	0.0141 (5)	0.0145 (5)	0.0019 (4)	0.0017 (4)	−0.0020 (4)
C4	0.0147 (5)	0.0137 (5)	0.0153 (5)	−0.0005 (4)	0.0011 (4)	−0.0033 (4)
C5	0.0167 (6)	0.0129 (5)	0.0149 (5)	0.0025 (4)	0.0020 (4)	0.0000 (4)
N6	0.0170 (5)	0.0129 (5)	0.0141 (5)	0.0028 (4)	0.0021 (4)	−0.0023 (4)
C7	0.0155 (5)	0.0136 (5)	0.0136 (5)	−0.0012 (4)	−0.0004 (4)	−0.0015 (4)
C8	0.0233 (6)	0.0133 (5)	0.0161 (6)	0.0023 (5)	0.0015 (5)	−0.0007 (4)
C9	0.0238 (6)	0.0157 (6)	0.0166 (6)	0.0020 (5)	0.0023 (5)	−0.0020 (4)
C10	0.0190 (6)	0.0162 (6)	0.0150 (6)	−0.0019 (5)	−0.0004 (4)	−0.0002 (4)
C11	0.0224 (6)	0.0136 (5)	0.0187 (6)	0.0015 (5)	−0.0010 (5)	0.0002 (4)
C12	0.0205 (6)	0.0133 (5)	0.0182 (6)	0.0025 (5)	0.0005 (5)	−0.0019 (4)
C13	0.0238 (6)	0.0185 (6)	0.0166 (6)	−0.0004 (5)	0.0008 (5)	0.0003 (5)
O14	0.0480 (7)	0.0236 (5)	0.0217 (5)	0.0104 (5)	0.0064 (4)	0.0063 (4)
O15	0.0336 (5)	0.0215 (5)	0.0143 (4)	0.0049 (4)	0.0051 (4)	0.0020 (3)
C16	0.0315 (7)	0.0260 (7)	0.0142 (6)	0.0027 (6)	0.0048 (5)	0.0024 (5)
C17	0.0338 (8)	0.0298 (7)	0.0214 (7)	0.0054 (6)	0.0058 (6)	−0.0011 (5)
O18	0.0223 (5)	0.0150 (4)	0.0201 (4)	0.0060 (3)	0.0021 (3)	−0.0024 (3)

Geometric parameters (Å, º)

S1—C2	1.7770 (12)	C9—H9	0.9500
S1—C5	1.7957 (12)	C10—C11	1.3918 (18)
C2—N3	1.3182 (15)	C10—C13	1.4915 (17)
C2—N6	1.3282 (15)	C11—C12	1.3811 (17)
N3—C4	1.3634 (15)	C11—H11	0.9500
C4—O18	1.2287 (15)	C12—H12	0.9500
C4—C5	1.5279 (16)	C13—O14	1.2054 (16)
C5—H5A	0.9900	C13—O15	1.3400 (16)
C5—H5B	0.9900	O15—C16	1.4470 (15)
N6—C7	1.4171 (15)	C16—C17	1.5081 (19)
N6—H6	0.859 (19)	C16—H16A	0.9900
C7—C8	1.3946 (17)	C16—H16B	0.9900
C7—C12	1.3977 (16)	C17—H17A	0.9800
C8—C9	1.3881 (17)	C17—H17B	0.9800
C8—H8	0.9500	C17—H17C	0.9800
C9—C10	1.3902 (17)		
C2—S1—C5	89.35 (5)	C9—C10—C11	119.57 (11)
N3—C2—N6	125.95 (11)	C9—C10—C13	121.63 (12)
N3—C2—S1	117.49 (9)	C11—C10—C13	118.79 (11)
N6—C2—S1	116.55 (9)	C12—C11—C10	120.38 (11)
C2—N3—C4	111.51 (10)	C12—C11—H11	119.8
O18—C4—N3	124.59 (11)	C10—C11—H11	119.8
O18—C4—C5	119.87 (10)	C11—C12—C7	119.75 (11)

N3—C4—C5	115.53 (10)	C11—C12—H12	120.1
C4—C5—S1	105.83 (8)	C7—C12—H12	120.1
C4—C5—H5A	110.6	O14—C13—O15	124.04 (12)
S1—C5—H5A	110.6	O14—C13—C10	124.54 (12)
C4—C5—H5B	110.6	O15—C13—C10	111.42 (11)
S1—C5—H5B	110.6	C13—O15—C16	116.64 (10)
H5A—C5—H5B	108.7	O15—C16—C17	106.36 (11)
C2—N6—C7	129.30 (10)	O15—C16—H16A	110.5
C2—N6—H6	115.7 (12)	C17—C16—H16A	110.5
C7—N6—H6	115.0 (12)	O15—C16—H16B	110.5
C8—C7—C12	120.37 (11)	C17—C16—H16B	110.5
C8—C7—N6	123.86 (11)	H16A—C16—H16B	108.6
C12—C7—N6	115.76 (11)	C16—C17—H17A	109.5
C9—C8—C7	119.11 (11)	C16—C17—H17B	109.5
C9—C8—H8	120.4	H17A—C17—H17B	109.5
C7—C8—H8	120.4	C16—C17—H17C	109.5
C8—C9—C10	120.81 (12)	H17A—C17—H17C	109.5
C8—C9—H9	119.6	H17B—C17—H17C	109.5
C10—C9—H9	119.6		

C5—S1—C2—N3	2.42 (10)	C7—C8—C9—C10	−0.3 (2)
C5—S1—C2—N6	−178.37 (10)	C8—C9—C10—C11	1.1 (2)
N6—C2—N3—C4	−178.45 (11)	C8—C9—C10—C13	−178.21 (12)
S1—C2—N3—C4	0.67 (14)	C9—C10—C11—C12	−0.94 (19)
C2—N3—C4—O18	176.49 (11)	C13—C10—C11—C12	178.37 (12)
C2—N3—C4—C5	−4.32 (15)	C10—C11—C12—C7	0.04 (19)
O18—C4—C5—S1	−174.96 (9)	C8—C7—C12—C11	0.75 (19)
N3—C4—C5—S1	5.82 (13)	N6—C7—C12—C11	179.87 (11)
C2—S1—C5—C4	−4.21 (8)	C9—C10—C13—O14	−179.18 (13)
N3—C2—N6—C7	1.3 (2)	C11—C10—C13—O14	1.5 (2)
S1—C2—N6—C7	−177.79 (10)	C9—C10—C13—O15	0.89 (18)
C2—N6—C7—C8	−8.0 (2)	C11—C10—C13—O15	−178.41 (11)
C2—N6—C7—C12	172.90 (12)	O14—C13—O15—C16	−0.5 (2)
C12—C7—C8—C9	−0.62 (19)	C10—C13—O15—C16	179.45 (11)
N6—C7—C8—C9	−179.66 (11)	C13—O15—C16—C17	172.17 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H6···O18^{i}	0.857 (17)	1.954 (17)	2.7882 (14)	164.3 (17)

Symmetry code: (i) *x*+1, *y*−1, *z*.