

Ethyl 2-amino-4-*tert*-butyl-1,3-thiazole-5-carboxylate and 6-methylimidazo[2,1-*b*]thiazole–2-amino-1,3-thiazole (1/1)

Daniel E. Lynch^{a*} and Ian McClenaghan^b

^aSchool of Science and the Environment, Coventry University, Coventry CV1 5FB, England, and ^bKey Organics Ltd, Highfield Industrial Estate, Camelford, Cornwall PL32 9QZ, England

Correspondence e-mail: apx106@coventry.ac.uk

Received 17 June 2004

Accepted 24 June 2004

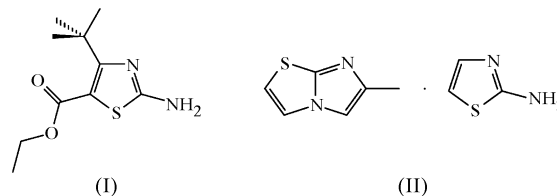
Online 21 July 2004

The structure of ethyl 2-amino-4-*tert*-butyl-1,3-thiazole-5-carboxylate, C₁₀H₁₆N₂O₂S, (I), and the structure of the 1:1 adduct 6-methylimidazo[2,1-*b*]thiazole–2-amino-1,3-thiazole (1/1), C₆H₆N₂S·C₃H₄N₂S, (II), have been determined. The molecules in (I) associate *via* a hydrogen-bonded $R_2^2(8)$ dimer consisting of N–H···N interactions, with the hydrogen-bonding array additionally involving N–H···O interactions to one of the carboxylate O atoms. The 2-aminothiazole molecules in (II) also associate *via* an N–H···N hydrogen-bonded $R_2^2(8)$ dimer, with an additional N–H···N interaction to the N_{sp²} atom of the imidazothiazole moiety, creating hydrogen-bonded quartets.

Comment

Aminothiazoles have been extensively studied for a range of biological and industrial applications (Lynch *et al.*, 1999; Toplak *et al.*, 2003). 2-Amino-1,3-thiazole, the structure of which was reported in 1982 (Caranoni & Reboul, 1982), is itself listed as a thyroid inhibitor (Merck, 2001). A search of the Cambridge Structural Database (CSD, Version 5.25 of April 2004; Allen, 2002) reveals that there are 73 crystal structures containing the 2-aminothiazole moiety, with 51 of those being pure organics. The present authors have recently published a paper on the packing modes of 2-amino-4-phenyl-1,3-thiazole derivatives (Lynch *et al.*, 2002) and have been investigating the structural aspects of 2-aminothiazole derivatives for the last six years. One such compound reported during this time was ethyl 4-*tert*-butyl-2-(3-phenylureido)-1,3-thiazole-5-carboxylate (Lynch & McClenaghan, 2002), which is currently the only structure of a 4-*tert*-butyl-5-ester derivative of an aminothiazole. However, we have recently determined the structure of ethyl 2-amino-4-*tert*-butyl-1,3-thiazole-5-carboxylate, (I), and report it here.

Another aminothiazole derivative is imidazo[2,1-*b*]thiazole, which has 11 analogues whose structures have previously been reported in the CSD. This bicyclic ring system can be prepared by refluxing a halomethyl ketone with 2-aminothiazole in ethanol. In an attempt to do so, using chloroacetone, an incomplete reaction yielded a mixture of the imidazo[2,1-*b*]thiazole derivative with the starting thiazole. The crystals that formed from the impure product were subsequently found to contain the 1:1 adduct of 6-methylimidazo[2,1-*b*]thiazole with 2-aminothiazole, (II), the structure of which is also reported here.



The structure of (I) consists of a single molecule (Fig. 1) which associates, *via* hydrogen-bonding interactions, to three symmetry-equivalent molecules (Fig. 2). One symmetry-

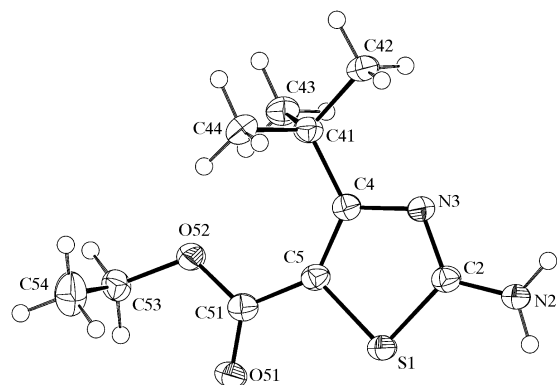


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

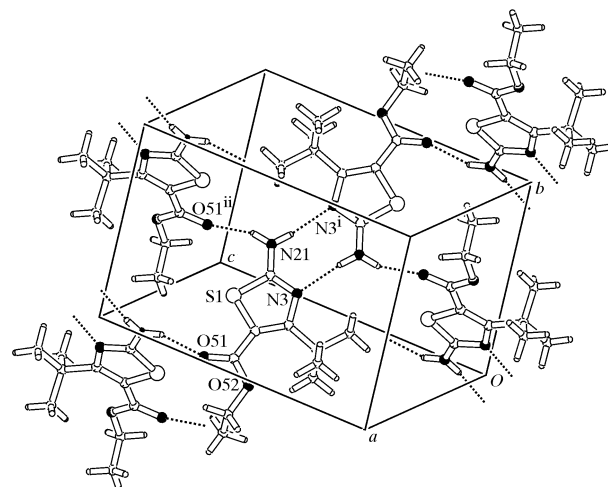


Figure 2
A packing diagram for (I). [Symmetry codes: (i) 1 - *x*, 1 - *y*, 1 - *z*; (ii) $\frac{3}{2} - x, y + \frac{1}{2}, \frac{3}{2} - z$.]

equivalent molecule forms a hydrogen-bonded $R_2^2(8)$ graph-set dimer (Etter, 1990) with (I) through an N—H...N interaction (Table 1), a feature common for 2-aminothiazole derivatives, while the other two associate to and from (I) through an N—H...O interaction. A similar packing mode has previously been observed in the structure of ethyl 2-amino-4-phenyl-1,3-thiazole-5-carboxylate (Lynch *et al.*, 2002), but is not observed in any other 5-ester-substituted 2-aminothiazole. This is probably due to the fact that, in each of these other structures, there are alternative exocyclic hydrogen-bonding acceptor atoms in addition to the two carboxylate O atoms. The ethyl chain twists out of the plane of the thiazole ring, with the C51—O52—C53—C54 torsion angle being $85.5(2)^\circ$, compared with $-168.5(3)^\circ$ in ethyl 4-*tert*-butyl-2-(3-phenylureido)-1,3-thiazole-5-carboxylate (Lynch & McClenaghan, 2002). One of the methyl groups in the *tert*-butyl moiety is aligned with the thiazole ring, with the N3—C4—C41—C42 torsion angle being $-1.8(2)^\circ$, similar to what was observed in ethyl 4-*tert*-butyl-2-(3-phenylureido)-1,3-thiazole-5-carboxylate [comparative torsion angle = $7.2(5)^\circ$].

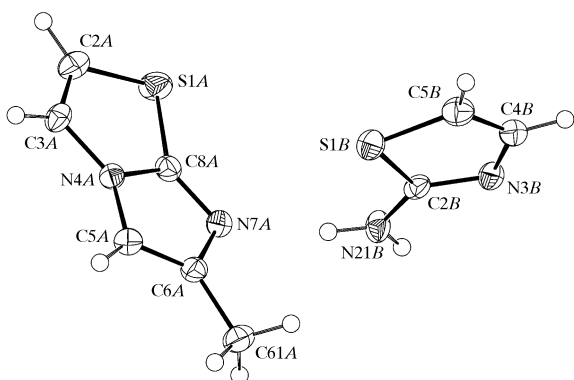


Figure 3
The molecular configuration and atom-numbering scheme for (II). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

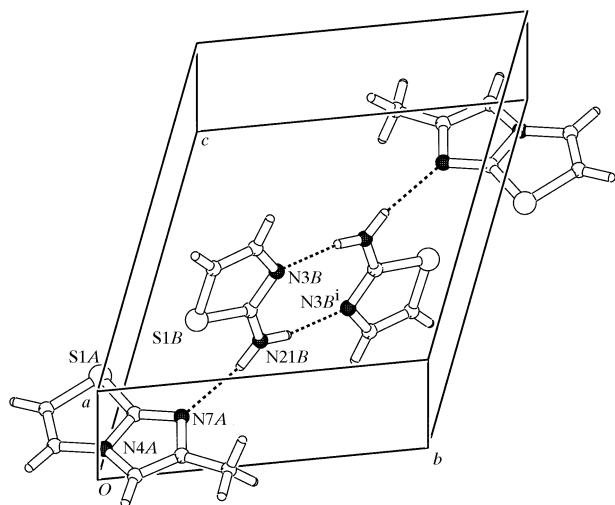


Figure 4
A packing diagram for (II). [Symmetry code: (i) $-x, 1 - y, 1 - z$.]

The structure of (II) comprises two adduct molecules associated by a single hydrogen-bonding interaction from one of the 2-amino H atoms to the N sp^2 atom in the imidazothiazole system (Fig. 3). Although one of the present authors (DEL) has determined 16 co-crystal structures containing 2-aminothiazole derivatives, there are only two previously reported co-crystals containing 2-aminothiazole itself (Kuz'mina & Struchkov, 1984; Moers *et al.*, 2000), and both of these are organic salts. The structure of (II) is unique in that it is the first adduct (not an organic salt) of 2-aminothiazole. The molecules in (II) pack across an inversion centre to construct an associated quartet, with the 2-aminothiazoles forming a hydrogen-bonded $R_2^2(8)$ graph-set dimer (Fig. 4). Hydrogen-bonding associations are listed in Table 2. A C—H...N close contact is also observed between atom C2A and the 2-amino N atom. The distance between atoms N7A and S1B is $3.294(3) \text{ \AA}$.

The determination of the structure of (II) and examination of the packing associations may now lead to a series of adducts containing 2-aminothiazole and heterocyclic bases, as opposed to continuing to try to obtain co-crystals (either adducts or organic salts) with organic acids.

Experimental

Compound (I) was obtained from Key Organics Ltd and was crystallized from ethanol. Compound (II) was prepared by refluxing equimolar amounts of 2-amino-1,3-thiazole and chloroacetone in ethanol for 16 h. Upon removal of the reaction solvent, the product was washed with aqueous NaOH and then extracted into dichloromethane. Crystals of (II) grew from the resultant liquid after removal of the extraction solvent.

Compound (I)

Crystal data

$C_{10}H_{16}N_2O_2S$
 $M_r = 228.31$
Monoclinic, $P2_1/n$
 $a = 10.6248(8) \text{ \AA}$
 $b = 8.6055(5) \text{ \AA}$
 $c = 13.0135(9) \text{ \AA}$
 $\beta = 92.977(4)^\circ$
 $V = 1188.24(14) \text{ \AA}^3$
 $Z = 4$

$D_x = 1.276 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 3289 reflections
 $\theta = 2.9\text{--}27.5^\circ$
 $\mu = 0.26 \text{ mm}^{-1}$
 $T = 120(2) \text{ K}$
Prism, colourless
 $0.42 \times 0.32 \times 0.08 \text{ mm}$

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SORTAV; Blessing, 1995)
 $T_{\min} = 0.913$, $T_{\max} = 0.977$
9227 measured reflections

2093 independent reflections
1668 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.082$
 $\theta_{\max} = 25.0^\circ$
 $h = -12 \rightarrow 12$
 $k = -10 \rightarrow 10$
 $l = -15 \rightarrow 15$

Table 1
Hydrogen-bonding geometry (\AA , $^\circ$) for (I).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
$N21\text{--}H21\cdots N3^i$	0.88	2.14	3.016(2)	173
$N21\text{--}H22\cdots O51^{ii}$	0.88	2.02	2.858(2)	158

Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$.

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.106$
 $S = 1.08$
 2093 reflections
 140 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0601P)^2 + 0.0328P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.20 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.34 \text{ e } \text{Å}^{-3}$

Compound (II)

Crystal data

$C_6H_6N_2S \cdot C_3H_4N_2S$
 $M_r = 238.33$
 Triclinic, $P\bar{1}$
 $a = 6.9195 (2) \text{ Å}$
 $b = 9.1860 (2) \text{ Å}$
 $c = 9.6953 (3) \text{ Å}$
 $\alpha = 69.5204 (17)^\circ$
 $\beta = 71.4823 (16)^\circ$
 $\gamma = 74.2770 (17)^\circ$
 $V = 538.48 (3) \text{ Å}^3$

$Z = 2$
 $D_x = 1.470 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 9410 reflections
 $\theta = 2.9\text{--}27.5^\circ$
 $\mu = 0.47 \text{ mm}^{-1}$
 $T = 120 (2) \text{ K}$
 Plate, colourless
 $0.26 \times 0.08 \times 0.04 \text{ mm}$

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SORTAV; Blessing, 1995)
 $T_{\min} = 0.885, T_{\max} = 0.982$
 12 593 measured reflections

2472 independent reflections
 2224 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.072$
 $\theta_{\max} = 27.6^\circ$
 $h = -8 \rightarrow 9$
 $k = -11 \rightarrow 11$
 $l = -12 \rightarrow 12$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.105$
 $S = 1.05$
 2472 reflections
 137 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.3904P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.26 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.44 \text{ e } \text{Å}^{-3}$

Table 2

Hydrogen-bonding geometry (Å, °) for (II).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N21B-H21B \cdots N3B^i$	0.88	2.14	3.010 (2)	170
$N21B-H22B \cdots N7A$	0.88	2.10	2.933 (2)	159
$C2A-H2A \cdots N21B^{ii}$	0.95	2.59	3.531 (2)	174

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

All H atoms were included in the refinement at calculated positions in the riding-model approximation, with N–H distances of 0.88 Å, and C–H distances of 0.95 (aromatic H atoms), 0.98 (CH₃ H atoms) and 0.99 Å (CH₂ H atoms), and with $U_{\text{iso}}(\text{H}) = 1.25U_{\text{eq}}(\text{C,N})$.

For both compounds, data collection: DENZO (Otwinowski & Minor, 1997) and COLLECT (Nonius, 1998); cell refinement: DENZO and COLLECT; data reduction: DENZO, SCALEPACK (Otwinowski & Minor, 1997) and COLLECT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLUTON94 (Spek, 1994) and PLATON97 (Spek, 1997); software used to prepare material for publication: SHELXL97.

The authors thank the EPSRC National Crystallography Service, Southampton, England.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1738). Services for accessing these data are described at the back of the journal.

References

Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
 Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–37.
 Caranoni, P. C. & Reboul, J. P. (1982). *Acta Cryst.* **B38**, 1255–1259.
 Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
 Kuz'mina, L. G. & Struchkov, Y. T. (1984). *Zh. Strukt. Khim.* **25**, 88–92. (In Russian.)
 Lynch, D. E. & McClenaghan, I. (2002). *Acta Cryst.* **E58**, o733–o734.
 Lynch, D. E., McClenaghan, I., Light, M. E. & Coles, S. J. (2002). *Cryst. Eng.* **5**, 123–136.
 Lynch, D. E., Nicholls, L. J., Smith, G., Byriel, K. A. & Kennard, C. H. L. (1999). *Acta Cryst.* **B55**, 758–766.
 Merck (2001). *The Merck Index*, 13th ed. New York: John Wiley & Sons.
 Moers, O., Wijaya, K., Lange, I., Blaschette, A. & Jones, P. G. (2000). *Z. Naturforsch. Teil B*, **55**, 738–752.
 Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
 Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
 Spek, A. L. (1994). *PLUTON94*. University of Utrecht, The Netherlands.
 Spek, A. L. (1997). *PLATON97*. University of Utrecht, The Netherlands.
 Toplak, R., Lah, N., Volmajer, J., Leban, I. & Le Maréchal, A. M. (2003). *Acta Cryst.* **C59**, o502–o505.

supporting information

Acta Cryst. (2004). C60, o592–o594 [doi:10.1107/S0108270104015471]

Ethyl 2-amino-4-*tert*-butyl-1,3-thiazole-5-carboxylate and 6-methylimidazo[2,1-*b*]thiazole–2-amino-1,3-thiazole (1/1)

Daniel E. Lynch and Ian McClenaghan

Computing details

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Nonius, 1998) for (I); *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998) for (II). For both compounds, cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO*, *SCALEPACK* (Otwinowski & Minor, 1997) and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLUTON94* (Spek, 1994) and *PLATON97* (Spek, 1997); software used to prepare material for publication: *SHELXL97*.

(I) Ethyl 2-amino-4-*tert*-butyl-1,3-thiazole-5-carboxylate

Crystal data

C₁₀H₁₆N₂O₂S

M_r = 228.31

Monoclinic, *P*2₁/*n*

Hall symbol: -*P* 2₁ *n*

a = 10.6248 (8) Å

b = 8.6055 (5) Å

c = 13.0135 (9) Å

β = 92.977 (4)°

V = 1188.24 (14) Å³

Z = 4

F(000) = 488

D_x = 1.276 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 3289 reflections

θ = 2.9–27.5°

μ = 0.26 mm⁻¹

T = 120 K

Prism, colourless

0.42 × 0.32 × 0.08 mm

Data collection

Bruker-Nonius KappaCCD area-detector diffractometer

Radiation source: Bruker Nonius FR591 rotating anode

Graphite monochromator

Detector resolution: 9.091 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)

T_{min} = 0.913, *T_{max}* = 0.977

9227 measured reflections

2093 independent reflections

1668 reflections with *I* > 2σ(*I*)

R_{int} = 0.082

θ_{max} = 25.0°, θ_{min} = 3.1°

h = -12→12

k = -10→10

l = -15→15

Refinement

Refinement on *F*²

Least-squares matrix: full

R [*F*² > 2σ(*F*²)] = 0.041

wR (*F*²) = 0.106

S = 1.08

2093 reflections

140 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0601P)^2 + 0.0328P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.34 \text{ e } \text{\AA}^{-3}$

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.73102 (5)	0.23587 (6)	0.65724 (3)	0.02690 (19)
C2	0.62597 (18)	0.3504 (2)	0.58627 (14)	0.0256 (5)
N21	0.57163 (16)	0.47329 (19)	0.62767 (12)	0.0318 (4)
H21	0.5186	0.5309	0.5900	0.040*
H22	0.5887	0.4969	0.6927	0.040*
N3	0.60506 (15)	0.30740 (18)	0.48900 (11)	0.0242 (4)
C4	0.67079 (18)	0.1747 (2)	0.46746 (14)	0.0233 (4)
C41	0.64797 (18)	0.1044 (2)	0.35997 (14)	0.0260 (5)
C42	0.5490 (2)	0.1988 (3)	0.29758 (15)	0.0366 (5)
H41	0.4713	0.2036	0.3348	0.046*
H42	0.5316	0.1491	0.2306	0.046*
H43	0.5807	0.3043	0.2873	0.046*
C43	0.7696 (2)	0.1048 (2)	0.30173 (15)	0.0326 (5)
H44	0.8007	0.2116	0.2964	0.041*
H45	0.7526	0.0622	0.2326	0.041*
H46	0.8333	0.0411	0.3389	0.041*
C44	0.5958 (2)	-0.0610 (2)	0.37003 (15)	0.0306 (5)
H47	0.6590	-0.1266	0.4064	0.038*
H48	0.5759	-0.1039	0.3013	0.038*
H49	0.5191	-0.0581	0.4088	0.038*
C5	0.74610 (18)	0.1188 (2)	0.54923 (14)	0.0249 (4)
C51	0.83058 (18)	-0.0117 (2)	0.56901 (14)	0.0261 (5)
O51	0.87845 (14)	-0.03732 (17)	0.65452 (10)	0.0345 (4)
O52	0.85479 (13)	-0.09931 (15)	0.48804 (9)	0.0288 (3)
C53	0.9321 (2)	-0.2367 (2)	0.50785 (16)	0.0312 (5)
H51	0.9964	-0.2144	0.5637	0.039*
H52	0.9764	-0.2640	0.4452	0.039*
C54	0.8522 (3)	-0.3706 (2)	0.5382 (2)	0.0484 (6)
H53	0.8094	-0.3440	0.6008	0.060*
H54	0.9055	-0.4621	0.5512	0.060*
H55	0.7893	-0.3933	0.4825	0.060*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0291 (3)	0.0302 (3)	0.0211 (3)	0.0021 (2)	-0.0018 (2)	-0.0009 (2)
C2	0.0268 (11)	0.0267 (10)	0.0232 (10)	-0.0036 (8)	-0.0002 (8)	0.0000 (8)
N21	0.0404 (11)	0.0325 (9)	0.0220 (8)	0.0094 (8)	-0.0049 (7)	-0.0022 (8)
N3	0.0277 (9)	0.0246 (8)	0.0203 (8)	-0.0001 (7)	-0.0004 (7)	-0.0006 (7)
C4	0.0235 (10)	0.0247 (10)	0.0219 (9)	-0.0045 (8)	0.0031 (8)	0.0015 (8)
C41	0.0264 (11)	0.0305 (11)	0.0213 (9)	0.0015 (8)	0.0012 (8)	0.0005 (8)

C42	0.0438 (14)	0.0413 (12)	0.0237 (10)	0.0090 (10)	-0.0068 (9)	-0.0071 (9)
C43	0.0385 (13)	0.0374 (12)	0.0225 (10)	0.0001 (10)	0.0071 (9)	0.0011 (9)
C44	0.0306 (12)	0.0337 (11)	0.0276 (10)	-0.0024 (9)	0.0029 (9)	-0.0067 (9)
C5	0.0272 (11)	0.0270 (10)	0.0208 (9)	-0.0024 (8)	0.0030 (8)	0.0006 (8)
C51	0.0255 (11)	0.0292 (11)	0.0239 (10)	-0.0035 (8)	0.0039 (8)	0.0021 (9)
O51	0.0395 (9)	0.0393 (9)	0.0241 (7)	0.0101 (7)	-0.0042 (6)	0.0016 (6)
O52	0.0315 (8)	0.0315 (8)	0.0233 (7)	0.0067 (6)	0.0017 (6)	-0.0011 (6)
C53	0.0293 (12)	0.0332 (11)	0.0315 (11)	0.0087 (9)	0.0046 (9)	0.0010 (9)
C54	0.0551 (16)	0.0331 (12)	0.0583 (15)	0.0020 (11)	0.0163 (13)	0.0017 (12)

Geometric parameters (Å, °)

S1—C2	1.7214 (19)	C43—H45	0.98
S1—C5	1.7434 (18)	C43—H46	0.98
C2—N3	1.327 (2)	C44—H47	0.98
C2—N21	1.332 (2)	C44—H48	0.98
N21—H21	0.88	C44—H49	0.98
N21—H22	0.88	C5—C51	1.452 (3)
N3—C4	1.375 (2)	C51—O51	1.219 (2)
C4—C5	1.384 (3)	C51—O52	1.332 (2)
C4—C41	1.532 (2)	O52—C53	1.455 (2)
C41—C42	1.529 (3)	C53—C54	1.496 (3)
C41—C43	1.532 (3)	C53—H51	0.99
C41—C44	1.535 (3)	C53—H52	0.99
C42—H41	0.98	C54—H53	0.98
C42—H42	0.98	C54—H54	0.98
C42—H43	0.98	C54—H55	0.98
C43—H44	0.98		
C2—S1—C5	88.99 (9)	H44—C43—H46	109.5
N3—C2—N21	123.57 (17)	H45—C43—H46	109.5
N3—C2—S1	115.09 (14)	C41—C44—H47	109.5
N21—C2—S1	121.34 (14)	C41—C44—H48	109.5
C2—N21—H21	120.0	H47—C44—H48	109.5
C2—N21—H22	120.0	C41—C44—H49	109.5
H21—N21—H22	120.0	H47—C44—H49	109.5
C2—N3—C4	111.38 (15)	H48—C44—H49	109.5
N3—C4—C5	114.28 (16)	C4—C5—C51	137.07 (18)
N3—C4—C41	117.14 (15)	C4—C5—S1	110.22 (14)
C5—C4—C41	128.52 (17)	C51—C5—S1	112.69 (13)
C42—C41—C4	110.29 (16)	O51—C51—O52	122.07 (17)
C42—C41—C43	108.08 (16)	O51—C51—C5	121.76 (18)
C4—C41—C43	110.68 (16)	O52—C51—C5	116.15 (16)
C42—C41—C44	107.26 (17)	C51—O52—C53	116.77 (14)
C4—C41—C44	109.21 (15)	O52—C53—C54	110.44 (17)
C43—C41—C44	111.25 (16)	O52—C53—H51	109.6
C41—C42—H41	109.5	C54—C53—H51	109.6
C41—C42—H42	109.5	O52—C53—H52	109.6

H41—C42—H42	109.5	C54—C53—H52	109.6
C41—C42—H43	109.5	H51—C53—H52	108.1
H41—C42—H43	109.5	C53—C54—H53	109.5
H42—C42—H43	109.5	C53—C54—H54	109.5
C41—C43—H44	109.5	H53—C54—H54	109.5
C41—C43—H45	109.5	C53—C54—H55	109.5
H44—C43—H45	109.5	H53—C54—H55	109.5
C41—C43—H46	109.5	H54—C54—H55	109.5
C5—S1—C2—N3	-1.16 (16)	C41—C4—C5—C51	2.1 (4)
C5—S1—C2—N21	179.29 (17)	N3—C4—C5—S1	1.1 (2)
N21—C2—N3—C4	-178.48 (18)	C41—C4—C5—S1	-176.03 (16)
S1—C2—N3—C4	2.0 (2)	C2—S1—C5—C4	0.00 (15)
C2—N3—C4—C5	-2.0 (2)	C2—S1—C5—C51	-178.60 (15)
C2—N3—C4—C41	175.51 (16)	C4—C5—C51—O51	-174.3 (2)
N3—C4—C41—C42	-1.8 (2)	S1—C5—C51—O51	3.8 (2)
C5—C4—C41—C42	175.29 (19)	C4—C5—C51—O52	7.1 (3)
N3—C4—C41—C43	117.77 (19)	S1—C5—C51—O52	-174.81 (13)
C5—C4—C41—C43	-65.2 (3)	O51—C51—O52—C53	5.6 (3)
N3—C4—C41—C44	-119.42 (18)	C5—C51—O52—C53	-175.82 (16)
C5—C4—C41—C44	57.7 (3)	C51—O52—C53—C54	85.5 (2)
N3—C4—C5—C51	179.2 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N21—H21...N3 ⁱ	0.88	2.14	3.016 (2)	173
N21—H22...O51 ⁱⁱ	0.88	2.02	2.858 (2)	158

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

(II) 6-Methylimidazo[2,1-*b*]thiazole 2-amino-1,3-thiazole

Crystal data

$C_6H_6N_2S \cdot C_3H_4N_2S$

$M_r = 238.33$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 6.9195$ (2) Å

$b = 9.1860$ (2) Å

$c = 9.6953$ (3) Å

$\alpha = 69.5204$ (17)°

$\beta = 71.4823$ (16)°

$\gamma = 74.2770$ (17)°

$V = 538.48$ (3) Å³

$Z = 2$

$F(000) = 248$

$D_x = 1.470$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9410 reflections

$\theta = 2.9$ – 27.5 °

$\mu = 0.47$ mm⁻¹

$T = 120$ K

Plate, colourless

$0.26 \times 0.08 \times 0.04$ mm

Data collection

Bruker-Nonius KappaCCD area-detector diffractometer

Radiation source: Bruker Nonius FR591 rotating anode

Graphite monochromator

Detector resolution: 9.091 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)

$T_{\min} = 0.885$, $T_{\max} = 0.982$

12593 measured reflections
 2472 independent reflections
 2224 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.072$

$\theta_{\text{max}} = 27.6^\circ$, $\theta_{\text{min}} = 3.2^\circ$
 $h = -8 \rightarrow 9$
 $k = -11 \rightarrow 11$
 $l = -12 \rightarrow 12$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.105$
 $S = 1.05$
 2472 reflections
 137 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.3904P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.26 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.44 \text{ e } \text{\AA}^{-3}$

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1A	0.00960 (8)	-0.08327 (6)	0.30204 (5)	0.03224 (15)
C2A	0.1187 (3)	-0.2132 (2)	0.1913 (2)	0.0286 (4)
H2A	0.1030	-0.3207	0.2263	0.036*
C3A	0.2265 (3)	-0.1461 (2)	0.0524 (2)	0.0243 (4)
H3A	0.2955	-0.1999	-0.0225	0.030*
N4A	0.2258 (2)	0.01230 (17)	0.03015 (16)	0.0202 (3)
C5A	0.3102 (3)	0.1371 (2)	-0.0801 (2)	0.0233 (4)
H5A	0.3949	0.1379	-0.1790	0.029*
C6A	0.2462 (3)	0.2595 (2)	-0.0171 (2)	0.0226 (4)
C61A	0.2951 (3)	0.4226 (2)	-0.0846 (2)	0.0301 (4)
H61A	0.3603	0.4435	-0.0190	0.038*
H62A	0.1670	0.4996	-0.0937	0.038*
H63A	0.3902	0.4316	-0.1855	0.038*
N7A	0.1235 (2)	0.21473 (18)	0.12958 (17)	0.0249 (3)
C8A	0.1169 (3)	0.0656 (2)	0.15266 (19)	0.0226 (4)
S1B	0.43793 (7)	0.20121 (5)	0.32915 (5)	0.02753 (15)
C2B	0.2352 (3)	0.3412 (2)	0.39595 (19)	0.0214 (3)
N21B	0.0592 (2)	0.38726 (19)	0.34887 (18)	0.0271 (3)
H21B	-0.0415	0.4574	0.3837	0.034*
H22B	0.0448	0.3473	0.2832	0.034*
N3B	0.2716 (2)	0.39372 (18)	0.49329 (17)	0.0240 (3)
C4B	0.4678 (3)	0.3223 (2)	0.5168 (2)	0.0256 (4)
H4B	0.5206	0.3462	0.5841	0.032*
C5B	0.5789 (3)	0.2182 (2)	0.4401 (2)	0.0294 (4)
H5B	0.7148	0.1621	0.4457	0.037*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1A	0.0402 (3)	0.0267 (3)	0.0227 (2)	-0.0067 (2)	-0.0004 (2)	-0.00468 (19)

C2A	0.0265 (9)	0.0212 (9)	0.0359 (10)	-0.0024 (7)	-0.0074 (8)	-0.0073 (8)
C3A	0.0220 (8)	0.0198 (8)	0.0329 (10)	-0.0007 (7)	-0.0073 (7)	-0.0115 (7)
N4A	0.0189 (7)	0.0199 (7)	0.0216 (7)	-0.0012 (5)	-0.0053 (5)	-0.0073 (6)
C5A	0.0209 (8)	0.0242 (9)	0.0224 (8)	-0.0043 (7)	-0.0031 (6)	-0.0058 (7)
C6A	0.0225 (8)	0.0205 (8)	0.0257 (9)	-0.0028 (6)	-0.0098 (7)	-0.0051 (7)
C61A	0.0314 (10)	0.0210 (9)	0.0369 (11)	-0.0039 (7)	-0.0116 (8)	-0.0052 (8)
N7A	0.0302 (8)	0.0223 (7)	0.0232 (7)	-0.0025 (6)	-0.0080 (6)	-0.0082 (6)
C8A	0.0244 (8)	0.0230 (8)	0.0198 (8)	-0.0021 (7)	-0.0059 (7)	-0.0069 (7)
S1B	0.0291 (3)	0.0251 (3)	0.0274 (3)	0.00164 (18)	-0.00392 (18)	-0.01401 (19)
C2B	0.0247 (8)	0.0162 (8)	0.0204 (8)	-0.0027 (6)	-0.0013 (6)	-0.0065 (6)
N21B	0.0271 (8)	0.0276 (8)	0.0316 (8)	0.0007 (6)	-0.0084 (6)	-0.0176 (7)
N3B	0.0262 (8)	0.0217 (7)	0.0246 (8)	-0.0019 (6)	-0.0052 (6)	-0.0102 (6)
C4B	0.0294 (9)	0.0230 (9)	0.0238 (9)	-0.0054 (7)	-0.0069 (7)	-0.0052 (7)
C5B	0.0272 (9)	0.0271 (9)	0.0308 (10)	-0.0011 (7)	-0.0069 (7)	-0.0077 (8)

Geometric parameters (Å, °)

S1A—C8A	1.7324 (18)	C61A—H62A	0.98
S1A—C2A	1.743 (2)	C61A—H63A	0.98
C2A—C3A	1.334 (3)	N7A—C8A	1.319 (2)
C2A—H2A	0.95	S1B—C5B	1.732 (2)
C3A—N4A	1.393 (2)	S1B—C2B	1.7518 (17)
C3A—H3A	0.95	C2B—N3B	1.312 (2)
N4A—C8A	1.363 (2)	C2B—N21B	1.345 (2)
N4A—C5A	1.381 (2)	N21B—H21B	0.88
C5A—C6A	1.368 (3)	N21B—H22B	0.88
C5A—H5A	0.95	N3B—C4B	1.391 (2)
C6A—N7A	1.388 (2)	C4B—C5B	1.340 (3)
C6A—C61A	1.495 (3)	C4B—H4B	0.95
C61A—H61A	0.98	C5B—H5B	0.95
C8A—S1A—C2A	89.74 (9)	H61A—C61A—H63A	109.5
C3A—C2A—S1A	113.09 (14)	H62A—C61A—H63A	109.5
C3A—C2A—H2A	123.5	C8A—N7A—C6A	104.09 (14)
S1A—C2A—H2A	123.5	N7A—C8A—N4A	112.88 (15)
C2A—C3A—N4A	111.98 (16)	N7A—C8A—S1A	136.17 (14)
C2A—C3A—H3A	124.0	N4A—C8A—S1A	110.93 (13)
N4A—C3A—H3A	124.0	C5B—S1B—C2B	89.06 (9)
C8A—N4A—C5A	106.52 (14)	N3B—C2B—N21B	124.61 (16)
C8A—N4A—C3A	114.26 (15)	N3B—C2B—S1B	114.35 (13)
C5A—N4A—C3A	139.17 (15)	N21B—C2B—S1B	121.04 (13)
C6A—C5A—N4A	105.51 (15)	C2B—N21B—H21B	120.0
C6A—C5A—H5A	127.2	C2B—N21B—H22B	120.0
N4A—C5A—H5A	127.2	H21B—N21B—H22B	120.0
C5A—C6A—N7A	110.99 (16)	C2B—N3B—C4B	109.87 (15)
C5A—C6A—C61A	128.64 (17)	C5B—C4B—N3B	116.87 (17)
N7A—C6A—C61A	120.35 (16)	C5B—C4B—H4B	121.6
C6A—C61A—H61A	109.5	N3B—C4B—H4B	121.6

C6A—C61A—H62A	109.5	C4B—C5B—S1B	109.85 (15)
H61A—C61A—H62A	109.5	C4B—C5B—H5B	125.1
C6A—C61A—H63A	109.5	S1B—C5B—H5B	125.1
C8A—S1A—C2A—C3A	-0.24 (15)	C3A—N4A—C8A—N7A	178.36 (14)
S1A—C2A—C3A—N4A	0.2 (2)	C5A—N4A—C8A—S1A	-178.08 (11)
C2A—C3A—N4A—C8A	0.0 (2)	C3A—N4A—C8A—S1A	-0.15 (19)
C2A—C3A—N4A—C5A	176.93 (19)	C2A—S1A—C8A—N7A	-177.8 (2)
C8A—N4A—C5A—C6A	-0.15 (18)	C2A—S1A—C8A—N4A	0.22 (14)
C3A—N4A—C5A—C6A	-177.26 (19)	C5B—S1B—C2B—N3B	-0.48 (14)
N4A—C5A—C6A—N7A	-0.16 (19)	C5B—S1B—C2B—N21B	179.75 (16)
N4A—C5A—C6A—C61A	178.67 (17)	N21B—C2B—N3B—C4B	-179.87 (17)
C5A—C6A—N7A—C8A	0.4 (2)	S1B—C2B—N3B—C4B	0.36 (19)
C61A—C6A—N7A—C8A	-178.53 (16)	C2B—N3B—C4B—C5B	0.0 (2)
C6A—N7A—C8A—N4A	-0.5 (2)	N3B—C4B—C5B—S1B	-0.4 (2)
C6A—N7A—C8A—S1A	177.48 (16)	C2B—S1B—C5B—C4B	0.45 (15)
C5A—N4A—C8A—N7A	0.4 (2)		

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N21 <i>B</i> —H21 <i>B</i> ...N3 <i>B</i> ⁱ	0.88	2.14	3.010 (2)	170
N21 <i>B</i> —H22 <i>B</i> ...N7 <i>A</i>	0.88	2.10	2.933 (2)	159
C2 <i>A</i> —H2 <i>A</i> ...N21 <i>B</i> ⁱⁱ	0.95	2.59	3.531 (2)	174

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