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2-Amino-5-butyl-4-methyl-1,3-thiazol-3-ium nitrate

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Key indicators

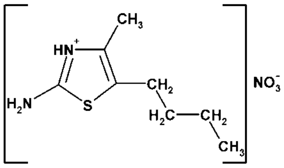
Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.131
Data-to-parameter ratio = 18.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_8H_{15}N_3O_3S$, shows bond lengths and angles that are typical and are in accordance with expected values. The structure comprises a substituted thiazolium ring that is connected to a nitrate ion *via* N—H···O hydrogen-bonding interactions.

Comment

The cation of the title compound, (I), is nearly planar. The torsion angles which deviate most significantly from 0 or 180° are S1—C5—C8—C9 [5.5 (3)°] and C4—C5—C8—C9 [−175.5 (2)°]. This corresponds to the twist of the butyl group with respect to the thiazole ring. The bond lengths and angles are in accordance with the expected values (Fitzsimons & Gallagher, 1999; Lynch & McClenaghan, 2001). Even the C2—S1—C5 angle of 90.9 (1)° is typical for substituted thiamines. A survey of the Cambridge Structural Database (Allen, 2002) for 1,3,4-thiazole rings gave a mean value of 90.8°.



(I)

Strong interactions are observed between the S atom and the nitrate ions. These interactions have been observed in other structures and it has been suggested that they have a mechanistic importance in C2-substituted thiamines (Yang *et al.*, 1987). This relatively strong short contact [S1···O15 = 3.15 (2) Å] contributes to the stabilization of the molecular packing along the *a* axis. The packing is stabilized by ionic interactions between the nitrate anions, the organic cations, and N—H···O hydrogen bonds. Two of the cations are joined head-to-head by the hydrogen bonds between them and the nitrate ions. These pairs of molecules form the characteristic chains extended along the *a* axis.

Experimental

To 50 ml of dry ether containing 0.5 g of $AlCl_3$ was added 2-heptanone (10 g, 87 mmol). The solution was cooled and 4.5 ml (0.087 mol) of bromine was added dropwise. HBr and ether were evaporated under a stream of dry nitrogen at 313 K. To the 6.7 g (88 mmol) of brown residue thiourea in 65 ml of water was added. The solution was warmed in a boiling water bath for 3 h. To the solution was then added 2 g of charcoal and 3.6 g (45 mmol) of NaOH. The solution was extracted with diethyl ether, diluted with petroleum ether and dried with $MgSO_4$. Crystals suitable for X-ray

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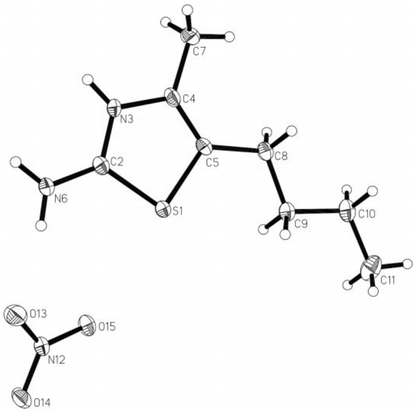

Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

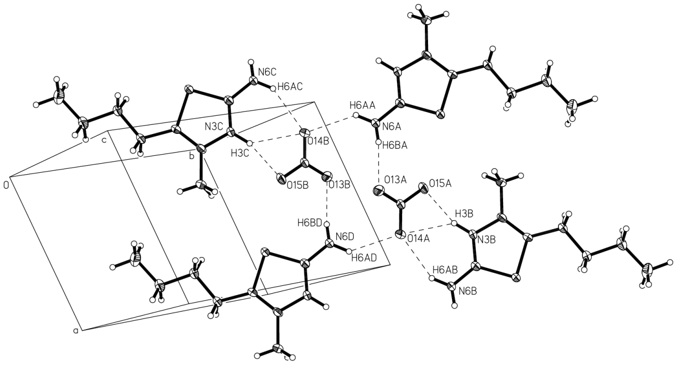

Figure 2
The crystal packing of the title compound. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (*A*) *x*, *y*, *z*; (*B*) 1 − *x*, *y*, *z*; (*C*) 1 − *x*, −*y*, −*z*; (*D*) −*x*, −*y*, −*z*.]

diffraction studies were grown by slow evaporation from *n*-heptane. 9.2 g (62.2%) of 2-amino-5-butyl-4-methylthiazole was obtained as light yellow flakes melting at 352–353 K. The title compound was prepared by dissolving 1.7 g (0.01 mol) of 2-amino-5-butyl-4-methylthiazole in 10 ml of methanol. Then, 0.76 ml (0.011 mol) of a 65% water solution of HNO_3 was added. 1.91 g (82%) of the product was filtered off and dried in a vacuum (m.p. 365–367 K). The products were recrystallized from methanol.

Crystal data

$C_8H_{15}N_3O_3S$
$M_r = 233.29$
Triclinic, $P\overline{1}$
$a = 7.486$ (2) Å
$b = 8.918$ (2) Å
$c = 9.240$ (2) Å
$\alpha = 76.26$ (3)°
$\beta = 87.15$ (3)°
$\gamma = 75.08$ (3)°
$V = 579.0$ (2) Å^3
$Z = 2$
$D_x = 1.338$ Mg m^{-3}
$D_m = 1.319$ Mg m^{-3}
D_m measured by flotation
Mo $K\alpha$ radiation
Cell parameters from 4395 reflections
$\theta = 3.6$–28.0°
$\mu = 0.27$ mm^{-1}
$T = 293$ (2) K
Irregular, colourless
0.20 × 0.15 × 0.10 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
ω scans
4395 measured reflections
2712 independent reflections
2016 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.056$
$\theta_{\rm max} = 28.0°$
$h = -10 \rightarrow 8$
$k = -12 \rightarrow 11$
$l = -12 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.131$
$S = 0.98$
2712 reflections
145 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0762P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.40$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.43$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—S1—C5	90.9 (1)	C8—C5—S1	121.7 (1)
C4—C5—C8	127.3 (2)		
C4—C5—C8—C9	−175.5 (2)	S1—C5—C8—C9	5.5 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H6*A*···O13	0.81 (3)	2.10 (3)	2.901 (3)	171 (2)
N3—H3*A*···O14$^{\rm i}$	0.92 (3)	2.36 (3)	3.090 (3)	135 (2)
N3—H3*A*···O15$^{\rm i}$	0.92 (3)	1.97 (3)	2.849 (3)	157 (2)
N6—H6*B*···O14$^{\rm i}$	0.90 (3)	2.25 (3)	3.045 (3)	146 (2)
N6—H6*B*···O14$^{\rm ii}$	0.90 (3)	2.25 (3)	2.994 (3)	138 (2)

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

H atoms bonded to C atoms were refined freely using a riding model. The coordinates of the H atoms bonded to N atoms were refined.

Data collection: *CrysAlisCCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlisCCD*; data reduction: *CrysAlisRED* (Oxford Diffraction, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL*97.

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