Crystal Structure of 2-Aminobenzothiazolinium Perchlorate

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The crystal structure of the title compound, $C_7H_7N_2S^+ClO_4^-$, contains cations and anions linked by N-H...O hydrogen bonds that form ribbons of alternating edge-fused $R_4^4(12)$ and $R_4^4(16)$ rings running along the *b* axis.

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Aminothiazoles and their derivatives belong to a group of biologically important compounds that have a very wide range of pharmacological activities, such as *e.g.* anti-inflammatory, immunoregulating, antiphlogistic-analgesics, antiulcer and psychotropic action.¹ They have also found applications in the treatment of allergies, hypertension, inflammation and schizophrenia, as well as bacterial and HIV infections.² This study was undertaken in order to understand the hydrogen-bonding patterns in 2-aminobenzothiazolinium perchlorate (Fig. 1).

The title compound was prepared by reaction of the equimolar quantities of quinoline and perchloric acid (65%). The resulting solid was recrystallized from water to afford crystals suitable for X-ray diffraction. The diffraction data were collected with KUMA KM4CCD diffractometer and CrysAlis CCD software.³ The structure was solved by a direct method using the SIR2002 program,⁴ and refined by a full-matrix least-squares method with anisotropic displacement parameters for the non-H atoms. All hydrogen atoms were located in a difference map, and their coordinates and isotropic displacement parameters were refined freely with resulting C-H distances in the range of 0.88(4)–0.95(3) Å. The crystal data and the experimental details are summarized in Table 1. The final fractional atomic coordinates and the equivalent isotropic displacement parameter for non-hydrogen atoms are listed in Table 2. Selected bond distances and bond angles are listed in Table 3, and the hydrogen-bonding geometric details are summarized in Table 4.

The presented title compound is composed of discrete 2-aminobenzothiazolinium cations and perchlorate anions (Fig. 2). 2-Aminobenzothiazolinium and perchlorate ions are connected together by N-H...O hydrogen bonds to form alternating edge-fused $R_4^4(12)$ and $R_4^4(16)$ rings. The combination of these motifs produces one-dimensional ribbons of $C_2^2(8)$ graph-set⁵ lying approximately in the (103) plane and running along the *b* axis, as shown in Fig. 3.

Fig. 1 Chemical structural diagram.

The 2-aminobenzothiazolinium moiety is not ideally planar. Only the six C atoms of the benzene ring are exactly coplanar. The N2, S1, C1 and C6 atoms are displaced from the five-membered ring plane by –0.030(4), –0.009(1), –0.010(1), and –0.009(1) Å, respectively.

The C1–S1 distance in the thiazole ring of 1.730(2) Å is intermediate between those reported for a single Csp³–S (1.819 Å) and a double C=S bond (1.61 Å), but longer than the C–S bond in thiophene (1.712 Å).⁷ The O atoms of the perchlorate anion are involved differently in the intermolecular hydrogen-

Table 1 Crystal and experimental data

Empirical formula	$C_7H_7N_2S^+ClO_4^-$
Formula weight	250.67
Temperature	296(2) K
Crystal size	0.42 × 0.28 × 0.07 mm
Crystal color	colorless
Crystal description	prism
Crystal system, space group, <i>Z</i>	triclinic, $P\bar{1}$, 2
<i>a</i> = 5.8021(3) Å	α = 85.805(4)°
<i>b</i> = 8.6623(4) Å	β = 77.486(4)°
<i>c</i> = 10.2595(5) Å	γ = 79.724(4)°
<i>V</i> = 494.99(4) Å ³	
Calculated density	1.682 g/cm ³
<i>F</i> (0 0 0)	256
Radiation	0.71073 Å (Mo- <i>K</i> α)
μ (Mo- <i>K</i> α)	0.592 mm ^{–1}
Absorption correction	multi-scan
Reflections collected / unique	5188 / 1936 [<i>R</i> _{int} = 0.0138]
2 θ _{max}	52.00°
Completeness to θ _{max}	99.8%
<i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> 1 = 0.0383, <i>wR</i> 2 = 0.1029
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0414, <i>wR</i> 2 = 0.1056
Goodness-of-fit	1.089
No. of reflections used	1936
No. of parameters	165
($\Delta\rho$) _{max}	< 0.001
($\Delta\rho$) _{max} , ($\Delta\rho$) _{min}	0.49, –0.35 e Å ^{–3}
Diffractometer	KUMA KM4CCD
Monochromator	graphite
Data collection program	CrysAlis CCD
Structure determination:	SIR2002
Structure refinement:	SHELXL97 ⁶
Structure drawing:	XSHELL and MERCURY
CCDC Deposit No.	284479

Table 2 Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$)

Atom	x	y	z	U_{eq}
S1	2023(1)	342(1)	7583(1)	45(1)
N1	1141(4)	3244(2)	8111(2)	39(1)
N2	-1957(4)	1883(3)	9127(2)	52(1)
C1	175(4)	1953(3)	8363(2)	38(1)
C2	4778(5)	4225(3)	6829(3)	48(1)
C3	6945(5)	3790(4)	5958(3)	58(1)
C4	7706(5)	2271(4)	5532(3)	61(1)
C5	6331(5)	1109(4)	5985(3)	52(1)
C6	4160(4)	1529(3)	6854(2)	39(1)
C7	3399(4)	3065(3)	7259(2)	36(1)
Cl	2352(1)	2763(1)	1475(1)	41(1)
O1	2748(6)	2508(3)	2782(2)	97(1)
O2	-110(4)	3358(3)	1494(4)	101(1)
O3	3664(5)	3914(3)	786(2)	85(1)
O4	3011(4)	1345(2)	749(2)	64(1)

$$U_{\text{eq}} = (1/3) \sum_i \sum_j \sum_k a_i^* a_j^* a_k^* a_i a_j a_k$$

Table 3 Selected bond distances ($\text{\AA}$) and angles ($^\circ$)

S1-C1	1.730(2)	S1-C6	1.759(2)
N1-C1	1.323(3)	N1-C7	1.398(3)
N2-C1	1.322(3)	Cl-O1	1.404(2)
Cl-O2	1.426(2)	Cl-O3	1.417(2)
Cl-O4	1.432(2)		
C1-S1-C6	90.16(10)	C1-N1-C7	115.1(2)
S1-C1-N1	112.46(17)	S1-C1-N2	123.2(2)
N1-C1-N2	124.3(2)	S1-C6-C5	128.5(2)
S1-C6-C7	110.72(16)	N1-C7-C2	126.7(2)
N1-C7-C6	111.5(2)		

Table 4 Hydrogen-bonding geometry ($\text{\AA}$, $^\circ$)

D-H...A	D-H	H...A	D...A	D-H...A
N1-H1...O2 ⁱ	0.72(3)	2.28(3)	2.937(3)	153(3)
N2-H6...O3 ⁱⁱ	0.83(4)	2.33(4)	3.069(4)	148(3)
N2-H7...O4 ⁱⁱⁱ	0.88(3)	2.09(3)	2.955(3)	166(3)

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

bond system. The Cl-O bonds involved in hydrogen-bonding are distinctly longer [1.417(2)–1.432(2) $\text{\AA}$] than the remaining not involved Cl-O1 bond [1.404(2) $\text{\AA}$]. A packing analysis does not indicate any significant stacking interactions or interactions with the S1 atom.

Supplementary material

Complete bond lengths and angles, coordinates and displacement parameters have been deposited at Cambridge Crystallography Data Centre (CCDC No. 284479).

References

1. Japan Patent, **1988**, JP63060978.

Fig. 2 View of the title compound with the atom numbering scheme. Displacement ellipsoids for non-hydrogen atoms are drawn at the 50% probability level.

Fig. 3 Packing diagram showing a ribbon of hydrogen-bonded (dashed lines) cations and anions running along the b axis.

2. P. C. Kearney, M. Fernandez, and J. A. Flygare, *J. Org. Chem.*, **1998**, 63, 196, and references therein.
3. Oxford Diffraction. CrysAlis CCD. Versions 1.171. Oxford Diffraction Ltd., Abingdon, Oxfordshire, England, **2004**.
4. M. C. Burla, M. Camalli, B. Carrozzini, G. L. Casarano, C. Giacovazzo, G., Polidori, and R. Spagna, *J. Appl. Cryst.*, **2003**, 36, 1103.
5. M. C. Etter, J. C. MacDonald, and J. Bernstein, *Acta Cryst. B*, **1990**, 46, 256.
6. G. M. Sheldrick, SHELXL97, A Program for Structure Determination, University of Göttingen, Germany, **1997**.
7. F. H. Allen, O. Kennard, D. G. Watson, L. Brammer, A. G. Orpen, and R. Taylor, *J. Chem. Soc. Perkin Trans. 2*, **1987**, 12, S1.