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A hydrogen-bonded supramolecular chain in thiabendazolium perchlorate

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Key indicators

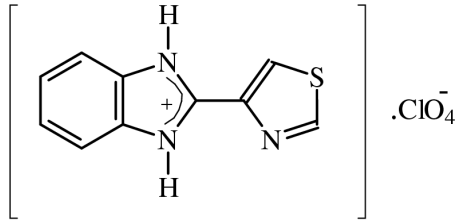
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.068
wR factor = 0.182
Data-to-parameter ratio = 10.8

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

In the title compound, thiabendazolium perchlorate [2-(4-thiazolyl)-1*H*-benzimidazol-1-ium perchlorate], $C_{10}H_8N_3S^+\cdot ClO_4^-$, one of the N atoms of the benzimidazole moiety is protonated rather than that in the thiazole group. This protonation leads to equalization of the bond angles at the two N atoms of the benzimidazole group. The C—C bond connecting the two ring systems has a length of 1.455 (7) Å. The dihedral angle between the benzimidazole system and the thiazole ring is 10.4 (3)°. The perchlorate anions bridge the thiabendazolium cations through a pair of N—H···O hydrogen bonds, leading to a hydrogen-bonded supramolecular chain.

Comment

Thiabendazole [2-(4-thiazolyl)-1*H*-benzimidazole] is a broad-spectrum anthelmintic, useful in the treatment of parasitic diseases in humans and animals. It is also useful as a fungicide for spoilage control of citrus fruit (Windholz, 1983). It is a bidentate chelating ligand, suggesting the involvement of metal chelation in its mode of action. The metal-chelating behaviour of thiabendazole is similar to that of 2,2′-bipyridine and 1,10-phenanthroline. The drug consists of two planar moieties, *viz*. benzimidazole and thiazole. The crystal structure of thiabendazole (Trus & Marsh, 1973) and its complexes with cobalt (Kowala & Wunderlich, 1973), copper (Udupa & Krebs, 1979) and platinum (Rong *et al.*, 1991) have been reported. The crystal structure of bis(thiabendazole)cobalt(II) chloride (Umadevi *et al.*, 1995), thiabendazolium chloride dihydrate and thiabendazolium bromide dihydrate (Prabakaran *et al.*, 2000), and thiabendazolium nitrate (Murugesan *et al.*, 1998) have also been reported from our laboratory. In all the crystal structures of the metal complexes of thiabendazole, the drug acts as a neutral bidentate chelator. One of the benzimidazole N atoms and the thiazole N atom are involved in the chelation. The thiazole S atom is not involved in coordination. The present study is aimed at understanding the conformation and hydrogen-bonding patterns of thiabendazolium perchlorate (TBPR), (I).



(I)

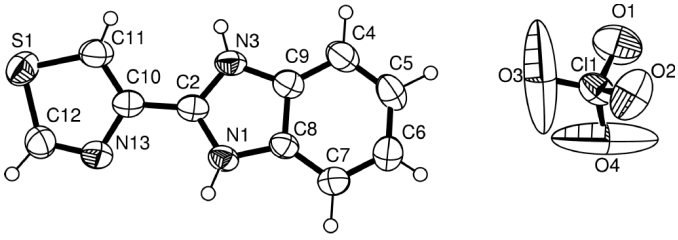


Figure 1
View of the title compound, with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

The Cl—O distances are in the range 1.368–1.404 Å, as expected (Muthiah *et al.*, 2002). An *ORTEP* view of TBPR is shown in Fig. 1. In this crystal structure, there is equalization of the bond angles at atoms N1 and N3 of the benzimidazole group, in contrast with the crystal structure of the free thiabendazole (Trus & Marsh, 1973). We have already reported the crystal structures of thiabendazolium bromide (Prabakaran *et al.*, 2000) and thiabendazolium chloride (Prabakaran *et al.*, 2000), in which the benzimidazole group is protonated. Therefore, we conclude that the benzimidazole group in (I) is also protonated. The two H atoms attached to N1 and N2 were geometrically fixed. The internal angle at the protonated atom N1, C2—N1—C8, is increased to 109.3 (4)°, compared with 103.8° in neutral thiabendazole (Trus & Marsh, 1973). The C—C bond connecting the two ring systems has a distance of 1.455 (7) Å; the corresponding value in neutral thiabendazole is 1.442 (1) Å. This distance is shorter than the ideal C—C bond length of 1.521 Å, suggesting appreciable partial-double-bond character in this bond (Trus & Marsh, 1973). The dihedral angle between the benzimidazole and thiazole ring systems is 10.4 (3)°, indicating that the thiabendazole moiety maintains a near planar geometry.

A view of the hydrogen-bonding pattern is shown in Fig. 2. The perchlorate anions bridge the thiabendazolium cations through a pair of N—H···O hydrogen bonds, leading to a hydrogen-bonded supramolecular chain. This type of supramolecular chain has also been observed in the crystal structure of thiabendazolium nitrate, where the nitrate anions bridge the thiabendazolium cations through N—H···O hydrogen bonds (Murugesan *et al.*, 1998).

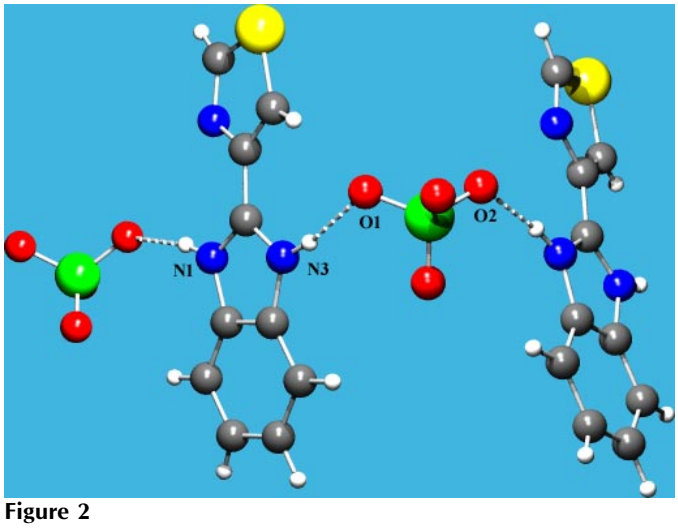


Figure 2
Hydrogen-bonding pattern in thiabendazolium perchlorate

Experimental

Thiabendazolium perchlorate was prepared by dissolving thiabendazole (obtained from Merck, Sharp & Dohme Inc., USA) in the minimum amount of dilute perchloric acid. The solution was warmed over a water bath for a few minutes. The resultant solution was allowed to cool slowly to room temperature. Colourless crystals were obtained after a few days.

Crystal data

$C_{10}H_8N_3S^+\cdot ClO_4^-$
M_r = 301.71
Monoclinic, $P2_1/a$
a = 14.121 (15) Å
b = 5.245 (2) Å
c = 17.384 (15) Å
β = 108.99 (11)°
V = 1217.4 (19) Å^3
Z = 4
D_x = 1.646 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–15°
μ = 0.50 mm^{-1}
T = 293 (2) K
Needle, colourless
0.30 × 0.30 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
2238 measured reflections
2136 independent reflections
1447 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 25.0°
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 6$
$l = -20 \rightarrow 19$
2 standard reflections
frequency: 60 min
intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.182
S = 1.22
2136 reflections
198 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 2.689P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.73 e Å^{-3}
$\Delta\rho_{min}$ = −0.63 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—O1	1.385 (5)	N1—C2	1.330 (6)
Cl1—O2	1.404 (5)	N1—C8	1.392 (6)
Cl1—O3	1.310 (9)	N3—C2	1.327 (6)
Cl1—O4	1.368 (8)	N3—C9	1.385 (6)
S1—C11	1.695 (5)	N13—C10	1.368 (7)
S1—C12	1.708 (7)	N13—C12	1.304 (7)
O1—Cl1—O2	112.1 (3)	N3—C2—C10	125.8 (4)
O1—Cl1—O3	107.3 (5)	N1—C2—N3	108.8 (4)
O1—Cl1—O4	103.4 (6)	N1—C8—C9	106.1 (4)
O2—Cl1—O3	110.6 (6)	N1—C8—C7	132.3 (5)
O2—Cl1—O4	108.5 (6)	N3—C9—C4	132.6 (5)
O3—Cl1—O4	114.8 (9)	N3—C9—C8	106.0 (4)
C11—S1—C12	89.0 (3)	N13—C10—C2	117.9 (5)
C2—N1—C8	109.3 (4)	N13—C10—C11	116.1 (5)
C2—N3—C9	109.8 (4)	S1—C11—C10	110.3 (4)
C10—N13—C12	109.1 (5)	S1—C12—N13	115.5 (4)
N1—C2—C10	125.3 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	2.04	2.881 (7)	166
N3—H3···O1	0.86	2.13	2.984 (8)	170
C5—H5···O3^{ii}	1.00 (5)	2.50 (5)	3.372 (9)	145 (5)
C7—H7···O3^{i}	1.00 (6)	2.48 (6)	3.336 (13)	143 (4)
C11—H11···O1	0.86 (6)	2.43 (5)	3.234 (8)	157 (4)

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

Atoms H1 and H3 were positioned geometrically and not refined. All other H atoms were located from a difference Fourier map and were refined isotropically.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 1990).

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