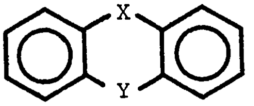
Table 4. *The central ring features of thioxanthene compared to similar ring systems*



X	*Y*	C–S	C–C	C–S–C	C–C–C	Angle of fold	Reference
S	S	1·77 (1) Å		100·3 (5)°		128·1°	Rowe & Post (1958)
S	S*	1·765 (2)		101·6 (1)		131·1	Wei (1971)
S	CH_2	1·77 (1)	1·51 (1) Å	99·2 (3)	111·4 (5)°	135·3 (1)	This work
SO	CHOH	1·77 (2)	1·50 (1)	97·5 (7)	112 (1)	132·4	Ternay, Chasar & Sax (1967)
SO	$CHCH_3$	1·782 (2)	1·521 (3)	95·2 (1)	108·8 (2)	126·0	Jackobs & Sundaralingam (1969)
CH_2	CH_2		1·53 (6)		111 (3)	143·8 (3)	Cordes & Malmstrom (1972)

* 2,7-Dimethyl derivative

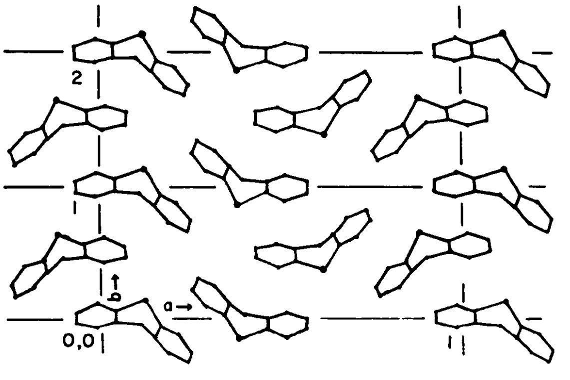


Fig. 2. The crystal packing of thioxanthene as viewed parallel to the *c* axis.

less than the corresponding van der Waals contact distances.

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Thiabendazole*

By Benes Louis Trus† and Richard E. Marsh

Arthur Amos Noyes Laboratory of Chemical Physics, California Institute of Technology, Pasadena, California 91109, *U.S.A.*

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Abstract. Crystals of the anthelmintic thiabendazole, 2-(4′-thiazolyl)benzimidazole ($C_{10}H_7N_3S$), are orthorhombic, space group *Pbca*, with $a=17{\cdot}052$ (7), $b=10{\cdot}998$ (4), and $c=10{\cdot}030$ (8) Å. There are eight formula units per cell; observed and calculated densities are 1·414 and 1·421 g cm^{-3}. Intensity data were collected on an automatic diffractometer; the structural parameters were refined by full-matrix least-squares to an *R* index of 0·066 for 1805 reflections. The two ring systems are approximately planar, but are twisted by 10° with respect to each other. The C–C bond connecting the two ring systems is 1·442 (10) Å long. Molecules are linked together by N(1)–H(1)···N(14) hydrogen bonds to form chains parallel to the *c* axis.

Introduction. Thiabendazole is a broad-spectrum anthelmintic useful in the treatment of the parasitic diseases of humans and other animals (Robinson, Phares

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& Graessle, 1964). Though its mode of operation is not known, its effects have been studied. Thiabendazole has been shown to be a good chelating agent (Robinson, Stoerk & Graessle, 1965), and to inhibit the rate of NADH oxidation (Prichard, 1970).

A sample of thiabendazole was provided by Dr Karst Hoogsteen of The Merck Institute for Therapeutic Research, Rahway, New Jersey. Crystals in the form of colorless needles elongated along **c** were prepared by evaporation of a methanol solution. The space group and approximate cell dimensions were obtained from Weissenberg photographs; more accurate cell constants were obtained by a least-squares fit to 2θ values measured on a diffractometer. The density was measured by flotation in a chloroform–ethanol mixture. Crystal data are given in Table 1.

Table 1. *Crystal data*

$C_{10}H_7N_3S$	F.W. 201·3
Space group, *Pbca*	Systematic absences:
$a = 17\cdot052$ (7) Å	$0kl$, $k = 2n+1$
$b = 10\cdot998$ (4)	$h0l$, $l = 2n+1$
$c = 10\cdot030$ (8)	$hk0$, $h = 2n+1$
$\lambda = 1\cdot5418$ Å	$Z = 8$
$V = 1881$ (3) Å^3	$D_c = 1\cdot421$ (2) g cm^{-3}
$\mu = 26\cdot3$ cm^{-1}	$D_m = 1\cdot414$ g cm^{-3}
$F(000) = 832$	

Intensity data were measured on a Datex-automated General Electric diffractometer, using Ni-filtered Cu $K\alpha$ radiation. The crystal had approximate dimensions $0\cdot19 \times 0\cdot23 \times 0\cdot39$ mm. Reflections with $2\theta \leq 150°$ were scanned in the θ–2θ mode at a rate of 2° (in 2θ) per min, the scan range varying from 2·95° at $2\theta = 6°$ to 4·17° at $2\theta = 150°$. A 30 sec background count was recorded on either side of the scan range. Two standard check reflections were measured after every 40 reflections; they showed no significant change in intensity.

A total of 1947 independent reflections were measured, of which 142 had negative values for their net intensities. Each reflection was assigned a variance $\sigma^2(I)$ based on counting statistics plus a term $(0\cdot02S)^2$, where S is the scan count (see for example Peterson & Levy, 1957). Intensities and their standard deviation $\sigma(I)$ were corrected for Lorentz and polarization factors, but not for absorption ($\mu r_{\max} \sim 0\cdot7$).

The structure was solved by the symbolic addition method (Sayre, 1952; Zachariasen, 1952; Karle & Karle, 1966; Long, 1965) based on the 190 reflections with $E \geq 1\cdot70$. The correct solution had the highest consistency (0·85) and required the fewest number of cycles to reach convergence. The resulting E map showed the location of all non-hydrogen atoms, which led to an R index $(= \sum|F_o - F_c|/\sum|F_o|)$ of 0·331 for reflections having $\sin^2\theta/\lambda^2 \leq 0\cdot2$. After three cycles of full-matrix least-squares adjustment of the coordinates and isotropic temperature factors ($R = 0\cdot174$), a difference map indicated the locations of the hydrogen atoms. Final refinement cycles included in a single matrix 156 parameters: coordinates for all 21 atoms, anisotropic thermal parameters for the 14 heavy atoms, isotropic B's for the 7 hydrogen atoms, a scale factor, and a secondary extinction factor (Larson, 1967). The final R index was 0·066, the weighted R $[= \sum w(|F_o|^2 - |F_c|^2)^2/\sum wF_o^4]$ 0·010, and the goodness-of-fit $\{= \sum w[(|F_o|^2 - |F_c|^2)^2/(M-S)]^{1/2}\}$ for $M = 1805$ observations and $S = 156$ parameters, 1·68. The largest shift in the final least-squares calculation was $0\cdot6\sigma$. A final difference map showed a maximum value of 0·29 e Å^{-3}, located near the S atom.

Scattering factors for C and N were from *International Tables for X-ray Crystallography* (1962), for S from Cromer & Waber (1965), and for H from Stewart, Davidson & Simpson (1965). The quantity minimized was $\sum w|F_o^2 - F_c^2|^2$, with weights w taken equal to $\sigma^{-2}(F_o^2)$ except for the 142 reflections having $I \leq 0$, for which $w = 0$. Computations were performed on an IBM 360-75 using the CRYM system (Duchamp, Westphal, Trus & Wang, unpublished programs). Fig. 1 was produced, in part, by *ORTEP* (Johnson, 1965).

Table 2. *Final atomic parameters and standard deviations* (*in parentheses*)

The anisotropic temperature coefficients are of the form

$$T = \exp\left[\tfrac{1}{4}(-h^2a^{*2}B_{11}\ldots - 2hka^*b^*B_{12}\ldots)\right].$$

Fractional coordinates are $\times 10^5$ for heavy atoms, $\times 10^4$ for hydrogen atoms. Anisotropic temperature coefficients are $\times 10^2$.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
N(1)	35361 (10)	76113 (19)	35657 (20)	347 (7)	359 (9)	212 (10)	1 (7)	−20 (8)	16 (8)
C(2)	39517 (11)	74281 (21)	24522 (22)	292 (7)	328 (9)	275 (10)	−41 (8)	5 (9)	−15 (9)
N(3)	36130 (10)	78509 (17)	13659 (17)	331 (7)	382 (9)	263 (8)	−20 (7)	−37 (7)	37 (7)
C(4)	29048 (11)	83500 (20)	18315 (24)	317 (8)	342 (9)	284 (11)	13 (7)	−35 (9)	0 (9)
C(5)	23188 (14)	89280 (23)	11300 (27)	438 (12)	434 (13)	315 (12)	77 (9)	−59 (9)	25 (9)
C(6)	16697 (15)	93419 (24)	18165 (31)	464 (12)	473 (13)	420 (16)	197 (10)	−57 (12)	16 (11)
C(7)	16406 (15)	91859 (26)	32032 (32)	398 (12)	547 (14)	503 (17)	170 (10)	76 (12)	−39 (12)
C(8)	22205 (15)	86200 (24)	38916 (31)	465 (12)	443 (13)	340 (13)	84 (10)	29 (10)	−13 (10)
C(9)	28520 (12)	82018 (19)	32000 (24)	333 (8)	330 (10)	313 (12)	12 (7)	−6 (9)	−4 (9)
C(10)	46977 (12)	68095 (19)	24583 (23)	289 (8)	332 (9)	355 (10)	−32 (7)	−9 (9)	2 (9)
C(11)	51936 (16)	66824 (29)	14133 (32)	432 (12)	665 (17)	482 (15)	123 (11)	120 (11)	212 (13)
S(12)	59752 (4)	58225 (8)	18496 (8)	438 (3)	729 (5)	646 (4)	222 (3)	184 (3)	157 (4)
C(13)	55960 (15)	56705 (26)	33889 (26)	437 (12)	524 (14)	434 (15)	128 (10)	−65 (11)	−2 (11)
N(14)	49351 (12)	62128 (20)	36024 (18)	411 (9)	514 (11)	306 (9)	96 (8)	−20 (8)	2 (9)

Table 2 (*cont.*)

	x	*y*	*z*	*B*(iso)
H(1)	3687 (14)	7425 (28)	4308 (28)	5·0 (0·8)
H(5)	2345 (12)	9091 (19)	127 (24)	4·4 (0·6)
H(6)	1368 (13)	9593 (22)	1448 (21)	2·3 (0·6)
H(7)	1270 (15)	9366 (24)	3620 (23)	4·7 (0·7)
H(8)	2183 (12)	8543 (20)	4804 (25)	4·1 (0·6)
H(11)	5142 (15)	7021 (23)	638 (28)	5·7 (0·8)
H(13)	5850 (18)	5186 (31)	4078 (33)	6·6 (1·0)

The final parameters are given in Table 2. The final value of the extinction parameter *g* (Larson, 1967, equation 3) was 2·3 (0·2) × 10^{-6}. A table of calculated and observed structure factors is available.*

Discussion. A drawing of the molecule is shown in Fig. 1. The thiazolyl and benzimidazole ring systems are each closely planar (Table 3). They are twisted, about the C(2)–C(10) bond, by 10° relative to one another; the C(2)–C(10) bond length of 1·442 (10) Å suggests appreciable delocalization across the bond. The protonated nitrogen atom, N(1), is *cis* to N(14) of the thiazolyl ring. This conformation may be stabilized by a weak electrostatic interaction H(1)· · ·N(14) (2·61 Å); a *trans* conformation would result in steric interference between H(1) and H(11).

Table 3. *Deviations from least-squares planes*

Plane 1 is the best plane through atoms N(1)-C(9) of the imidazole ring, all weighted equally; plane 2 is the best plane through C(10)-N(14) of the thiazolyl ring.

	Deviation Plane 1*	Deviation Plane 2†
N(1)	0·003 Å	0·081 Å
C(2)	0·000	−0·064
N(3)	0·001	−0·284
C(4)	−0·004	−0·289
C(5)	0·006	−0·463
C(6)	−0·006	−0·431
C(7)	0·005	−0·190
C(8)	0·000	−0·014
C(9)	−0·006	−0·066
C(10)	−0·018	0·003
C(11)	0·105	−0·003
S(12)	−0·062	0·002
C(13)	−0·298	−0·001
N(14)	−0·258	−0·001
H(1)	0·04	0·26
H(5)	0·05	−0·59
H(6)	−0·05	−0·56
H(7)	−0·05	−0·21
H(8)	0·02	0·15
H(11)	0·29	0·03
H(13)	−0·48	−0·02

* Direction cosines of plane normal: 0·4559, 0·8801, 0·1329; origin-to-plane distance, 10·588 Å.

† Direction cosines of plane normal: 0·4906, 0·8210, 0·2921; origin-to-plane distance, 10·796 Å.

Bond distances, and those bond angles that involve only the heavier atoms, are included in Fig. 1; bond angles involving hydrogen atoms are listed in Table 4. The formal e.s.d.'s in the distances are about 0·003 Å for the C–S bonds, 0·004 Å for C–C and C–N, and 0·03 Å for C–H and N–H. However, we view these e.s.d.'s with some skepticism, because of a number of rather distressing peculiarities associated with our final model: (1) the C(6)–H(6) and C(7)–H(7) distances are very short, even by X-ray standards; (2) other distances and angles in the benzene ring, and particularly those involving C(6) and C(7), do not show the approximate twofold symmetry that one might expect; (3) atoms C(11) and S(12) of the thiazolyl ring show extreme 'temperature' anisotropies, suggesting out-of-plane displacements of r.m.s. amplitude about 0·36 Å. It is, of course, quite possible that the large thermal displacements of atoms C(11) and S(12) represent a disorder involving two non-planar conformations of the thiazolyl ring; such a model would explain the rather short values found for the C–S bond distances. The peculiarities associated with C(6) and C(7) are more difficult to rationalize; while the anisotropic thermal parameters of these atoms are rather large (maximum r.m.s. displacement 0·29 Å), they are not large enough to suggest a simple explanation for either the shortening of the C–H bonds or the asymmetry in the ring geometry. In view of these anomalies, we believe that more

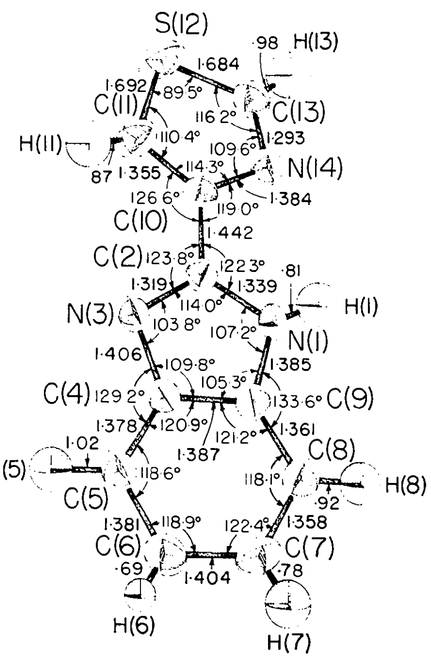

Fig. 1. The thiabendazole molecule. The atoms are represented by temperature-factor ellipsoids drawn at the 40 % probability level (Johnson, 1965). Estimated standard deviations are about 0·5° in the bond angles, 0·01 Å in distances between pairs of heavy atoms, and 0·1 Å in distances involving hydrogen atoms.

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* This table has been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30145 (11pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

realistic values for the standard deviations are about 0·01 Å for bonds involving C, N, and S, and 0·10 Å for the C–H and N–H bonds.

Table 4. *Bond angles involving hydrogen atoms*

Estimated standard deviations are about 3°.

H(1)—N(1)—C(2)	124°	H(1)—N(1)—C(9)	129°
H(5)—C(5)—C(4)	124	H(5)—C(5)—C(6)	118
H(6)—C(6)—C(5)	117	H(6)—C(6)—C(7)	124
H(7)—C(7)—C(6)	122	H(7)—C(7)—C(8)	116
H(8)—C(8)—C(7)	120	H(8)—C(8)—C(9)	122
H(11)-C(11)-S(12)	124	H(11)-C(11)-C(10)	126
H(13)-C(13)-S(12)	122	H(13)-C(13)-N(14)	121

Views of the structure, as seen down the *b* and *c* axes, are shown in Figs. 2 and 3. The predominating intermolecular interactions are N(1)-H(1)···N(3) bonds with dimensions N···N 2·86 Å, H···N 2·10 Å, and N-H···N 156°. These hydrogen bonds link molecules together in a criss-cross fashion (Fig. 3) to form a column, running along **c**, that is perhaps vaguely suggestive of a cheval-de-frise. Adjacent columns pack together relatively loosely; the shortest intermolecular distance between heavy atoms is a C(13)···N(3) contact of 3·39 Å between molecules related by a 2_1 axis along **b**. Although there is some suggestion of parallel stacking of benzene and imidazole rings along the [110] (and [1$\bar{1}$0]) direction, the dihedral angle between the rings is 25° and the shortest contact distances are S···C(8), 3·74 Å and C(11)···C(7), 3·72 Å.

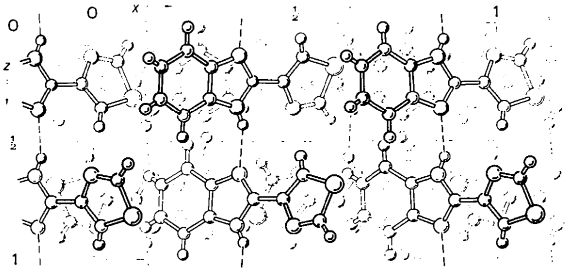

Fig. 2. A drawing of the structure, viewed down the *b* axis.

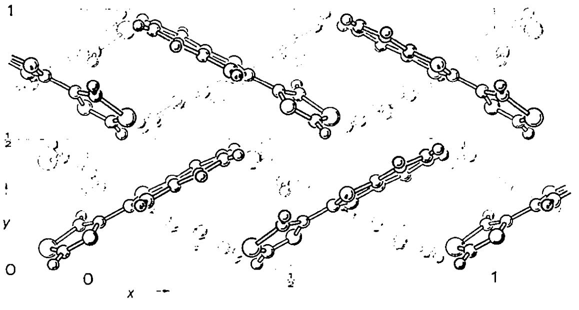

Fig. 3. A drawing of the structure, viewed down the *c* axis.

The nitrogen atom of the thiazolyl ring, N(14), is not involved in a hydrogen bond. One might have anticipated that N(3) would be a better hydrogen-bond acceptor than N(14), for by accepting a hydrogen bond, N(3) assumes an electronic configuration approaching that of N(1), increasing the symmetry and, hence, the resonance stabilization of the imidazole ring.

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