the anomalous dispersion scattering factor for Zr is from Cromer (1965). The structure was determined by iterative least-square adjustment with a modification of *ORFLS* (Busing, Martin & Levy, 1962). The position of the Li ion was easily determined from a difference map.

Discussion. The structure proposed by Hoppe & Dahne (1960) is essentially correct and the Li ions are on positions 2(*d*); $\frac{1}{3},\frac{2}{3},\frac{1}{2}$. The atomic parameters are listed in Table 1.[†] Figs. 1 and 2 are respectively one asymmetrical unit and the lithium coordination polyhedra surrounding one Zr ion, both views parallel to **c**. Each Zr^{4+} is coordinated by $6F^-$ at the corners of a regular octahedron; Zr–F = 2·016 (1) Å. Similarly each Li^+ is coordinated by $6F^-$ at 2·0246 (9) Å. Each corner of the ZrF_6^{2-} ion is shared by two Li^+ octahedra that each share edges with one another, as illustrated by Fig. 3, with a total of $12Li^+$ ions coordinating each ZrF_6^{2-} ion; Li–Zr = 3·6971 (5) Å.

Table 1. *Atomic and thermal parameters* (× 10^4)

$\varrho_x = 3·646$ g cm^{-3}, $R(F_o^2) = 0·0605$

Coefficients in the temperature factor:

$$\exp\,[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)]\,.$$

The number in parentheses is the standard error in terms of the last significant digit as derived from the variance-covariance matrix.

	Li	Zr	F
Wyckoff notation	2(*d*)	1(*a*)	6(*k*)
x	$\frac{1}{3}$	0	0·6724 (3)
y	$\frac{2}{3}$	0	0
z	$\frac{1}{2}$	0	0·2550 (3)
β_{11}	206 (20)	121 (1)	180 (3)
β_{22}	β_{11}	β_{11}	195 (5)
β_{33}	200 (24)	95 (1)	163 (3)
β_{12}	$\frac{1}{2}\beta_{11}$	$\frac{1}{2}\beta_{11}$	$\frac{1}{2}\beta_{22}$
β_{13}	0	0	30 (3)
β_{23}	0	0	0

References

Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS*. USAEC Report ORNL-TM-305, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Cromer, D. T. (1965). *Acta Cryst.* **18**, 17–23.

Cromer, D. T. & Waber, J. T. (1965). *Acta Cryst.* **18**, 104–109.

Hoppe, R. & Dahne, W. (1960). *Naturwissenschaften*, **47**, 397.

---

[*] Research sponsored by U.S. Atomic Energy Commission under contract with the Union Carbide Corporation.

[†] A table of structure factors has been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30154. Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Acta Cryst. (1973). B**29**, 2296

Thioxanthene

By J. A. Gillean III, D. W. Phelps and A. W. Cordes

Department of Chemistry, University of Arkansas, Fayetteville, Arkansas 72701, *U.S.A.*

(*Received* 10 *May* 1973; *accepted* 7 *June* 1973)

Abstract. $C_{13}H_{10}S$, orthorhombic, $P2_12_12_1$, $a = 21·096$ (4), $b = 7·804$ (1), $c = 6·049$ (1) Å, $Z = 4$, $D_c = 1·326$, $D_x = 1·33$ g cm^{-3} (by suspension). The C–S bond lengths are 1·781 (6) and 1·759 (6) Å, the CSC angle is 99·2 (3)°, and the angle between the phenylene planes is 135·3 (1)°.

Introduction. Slow evaporation of thioxanthene–methylene chloride–octane solution yielded colorless crystals. The crystal selected for data collection was bounded by the faces {100}, {110}, {$\bar{1}$10}, and {001}. Distances from the center of mass to these face planes were 0·36, 0·22, 0·27, and 0·36 mm (all ± 0·01 mm), respectively. The crystal was mounted with **c** parallel to the φ axis of the diffractometer. Unit-cell parameters and intensity data were obtained on a G. E. XRD-5 manually operated quarter-circle diffractometer with Zr-filtered Mo $K\alpha$ ($\lambda = 0·71069$ Å) radiation and a scintillation detection system. Systematic absences of *h*00 with *h* odd, 0*k*0 with *k* odd, 00*l* with *l* odd indicated space group $P2_12_12_1$ (No. 19). No absorption or extinction corrections were made. The linear absorption coefficient is 2·72 cm^{-1}; calculated transmission coefficients for *F* varied from 0·97 to 0·94.

Data collection involved θ–2θ scans of 2° at 2°/min, with 10-sec stationary backgrounds at each end of the scans. All 1062 unique reflections with $0° < 2\theta < 50°$ were examined; 768 had $I \geq 2\sigma(I)$ and these were used for the refinement. Four reflections measured periodically during the two days of data collection varied by less than 2σ and thereby indicated crystal and electronic stability.

The standard deviations for *I* were calculated by $\sigma(I) = (S + 9B + 0·0016I^2)^{1/2}$ (where S = scan count, B = sum of the background counts, and $I = S - 3B$), except for a small number of reflections for which the two background readings differed by more than twice the calculated σ owing to tailing of the reflection peak. For the latter reflections the standard deviations were set equal to the deviation of the background readings from the mean background value.

Neutral atom scattering factors were used (*International Tables for X-ray Crystallography*, 1973), and real and imaginary anomalous dispersion corrections were made for sulfur (Cromer, 1965).

The trial structure was initiated by Patterson methods. Least-squares refinement with isotropic temperature factors for all atoms (calculated hydrogen positions based on C–H = 0·95 Å were included for structure-factor calculations but were not refined) gave $R=7{\cdot}2$ and $R_w=7{\cdot}8$. With anisotropic thermal parameters for sulfur $R=5{\cdot}8$ and $R_w=6{\cdot}4$; this change is significant on the 99·5% level (Hamilton, 1965). The standard deviation of an observation of unit weight was 1·60, and the final difference Fourier map had a maximum peak of 0·27 e Å^{-3}(=4% of a carbon peak). Atomic positions and thermal parameters are given in Tables 1 and 2.* Fig. 1 displays the bond distances, bond angles, and their e.s.d.'s. Fig. 2 shows the packing in the unit cell.

Table 1. *Fractional atomic coordinates and isotropic thermal parameters for thioxanthene*

	x	*y*	*z*	*B* (Å²)
S	0·1177 (1)	0·1222 (2)	0·2130 (3)	–
C(1)	0·0443 (3)	0·0563 (7)	0·056 (1)	3·1 (1)
C(2)	−0·0114 (3)	0·0979 (8)	0·207 (1)	4·2 (1)
C(3)	−0·0680 (3)	0·0473 (9)	0·116 (1)	4·7 (2)
C(4)	−0·0700 (3)	−0·0408 (8)	−0·083 (1)	4·7 (2)
C(5)	−0·0138 (3)	−0·0819 (7)	−0·190 (1)	4·1 (1)
C(6)	0·0446 (3)	−0·0316 (7)	−0·104 (1)	3·2 (1)
C(7)	0·1060 (3)	−0·0680 (7)	−0·220 (1)	3·5 (1)
C(8)	0·1556 (3)	−0·1370 (7)	−0·061 (1)	3·1 (1)
C(9)	0·1944 (3)	−0·2738 (8)	−0·112 (1)	3·8 (1)
C(10)	0·2423 (3)	−0·3263 (9)	0·029 (1)	4·4 (1)
C(11)	0·2504 (3)	−0·2454 (8)	0·231 (1)	4·0 (1)
C(12)	0·2116 (3)	−0·1090 (8)	0·245 (1)	3·7 (1)
C(13)	0·1644 (3)	−0·0557 (7)	0·141 (1)	3·0 (1)
H(C2)	−0·0106	0·1594	0·341	5·0
H(C3)	−0·1063	0·0746	0·191	5·0
H(C4)	−0·1096	−0·0746	−0·144	5·0
H(C5)	−0·0145	−0·1435	−0·326	5·0
H(C7)	0·1214	0·0352	−0·284	5·0
H(C7)	0·0989	−0·1501	−0·333	5·0
H(C9)	0·1885	−0·3310	−0·249	5·0
H(C10)	0·2693	−0·4191	−0·009	5·0
H(C11)	0·2826	−0·2821	0·330	5·0
H(C12)	0·2175	−0·0519	0·423	5·0

Table 2. *Anisotropic thermal parameters of the sulfur atom in thioxanthene*

Thermal parameters are of the form

$$\exp\left[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)\right].$$

The β_{ij}'s are multiplied by 10^4.

β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
23 (1)	171 (3)	236 (5)	1 (1)	−4 (1)	52 (4)

Discussion. The χ^2 values for the two C_6SC groups in thioxanthene (Table 3) indicate non-planarity; however, the deviations from planarity are less than 0·03 Å and the significance of the displacements of C(7) and S from the best plane is no greater than that of the carbon atoms of the phenylene rings. Table 4 compares the angles and distances of the S and C(7) atoms with similar parameters of analogous molecules whose structures have been reported. This comparison shows that the C–S distances and C–C (central ring) distances in these heterocyclic molecules are generally insensitive to varying oxidation states (S and SO) and substitutions on the central carbon atom. These distances indicate little, if any, π interactions with the phenylene groups. Table 4 shows that the bond angles at these central atoms are more responsive to these chemical changes, and the angles of fold on the $X\cdots Y$ line of the two molecular 'halves' are obviously the most widely varying structural parameter in the series.

The structure of thioxanthene, together with those of thianthrene ($C_6H_4\langle{}^{S}_{S}\rangle C_6H_4$) and dihydroanthracene ($C_6H_4\langle{}^{CH_2}_{CH_2}\rangle C_6H_4$), provide the first such series for this type of non-planar heterocyclic molecule. It will be of interest to see if similar sets of these ring systems show the linear variation in the angle of fold displayed by this series.

Table 3. *Distances to best planes in thioxanthene*

All atoms listed for each plane were included in the calculation of the plane; weighting was proportional to $1/\sigma^2_\perp$, where $\sigma_\perp$ is the positional e.s.d. normal to the plane.

S	0·001 (1) Å	S	−0·001 (1) Å
C(7)	−0·018 (6)	C(7)	−0·026 (5)
C(1)	0·007 (6)	C(8)	−0·028 (6)
C(2)	−0·012 (6)	C(9)	0·000 (6)
C(3)	−0·008 (7)	C(10)	−0·016 (6)
C(4)	−0·006 (7)	C(11)	−0·028 (7)
C(5)	0·019 (6)	C(12)	0·021 (6)
C(6)	0·007 (6)	C(13)	0·028 (6)
$\chi^2=29{\cdot}5$		$\chi^2=107$	

Fig. 2 shows the relatively simple crystal packing of thioxanthene. There are no intermolecular separations

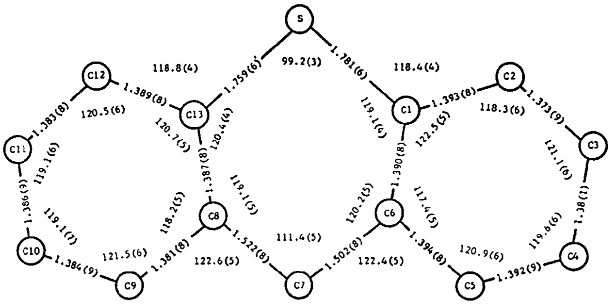
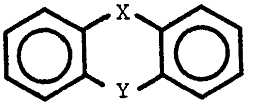


Fig. 1. Bond distances and bond angles within the thioxanthene molecule.

---

* A list of structure factors has been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30155 (5 pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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

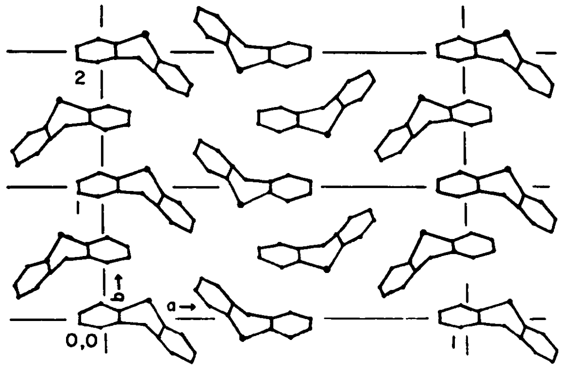


Fig. 2. The crystal packing of thioxanthene as viewed parallel to the *c* axis.

less than the corresponding van der Waals contact distances.

We would like to thank the donors of the Petroleum Research Foundation, administered by the American Chemical Society, and the National Science Foundation's Undergraduate Research Participation Program.

References

Cordes, A. W. & Malmstrom, M. C. (1972). Unpublished results.

Cromer, D. T. (1965). *Acta Cryst.* **18**, 17–23.

Hamilton, W. C. (1965). *Acta Cryst.* **18**, 502–510.

International Tables for X-ray Crystallography (1973). Vol. IV. In the press.

Jackobs, J. & Sundaralingam, M. (1969). *Acta Cryst.* **B25**, 2487–2496.

Rowe, I. & Post, B. (1958). *Acta Cryst.* **11**, 372–374.

Ternay, A. L. Jr, Chasar, D. W. & Sax, M. (1967). *J. Org. Chem.* **32**, 2465–2470.

Wei, C. H. (1971). *Acta Cryst.* **B27**, 1523–1527.

Acta Cryst. (1973). **B29**, 2298

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.*

(*Received* 2 *April* 1973; *accepted* 23 *May* 1973)

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

---

* Contribution No. 4668 from the Arthur Amos Noyes Laboratory of Chemical Physics. This investigation was supported in part by Public Health Service Research Grant No. GM 16966 from the National Institute of General Medical Sciences, National Institutes of Health.

† Supported in part by National Institute of General Medical Sciences Research Training Grant 5 TO1 GM-01262-08-BCH.