Talipov, Saibova & Aripov, 1981), significant differences are observed mainly for the C—O and C—N distances; these authors report C—O distances of 1·427 and 1·430 Å and C—N distances of 1·496 and 1·506 Å for the two independent groups while 1·415 and 1·470 Å are observed in the title compound.

As shown in Fig. 1, a projection along the *b* axis, the structure can be described as a succession of layers perpendicular to the *c* axis. These layers contain both the P_4O_{12} ring anions and the organic groups and are separated by a distance of *c*/2.

A three-dimensional network of hydrogen bonds, whose geometrical details are reported in Table 2, connects the layers and the organic groups and P_4O_{12} anions within each layer.

As normally observed the bonding O atoms [O(*L*)] do not take part in the hydrogen-bond network.

Fig. 2 shows a projection along the *c* axis.

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Structure of $C_{22}H_{36}N_{10}O_2S_2$

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Abstract. Methyl 3,3-dimethyl-2-oxobutyrate, 2-(*Z*,*Z*)-azine with 2-[(1*E*,3*Z*)-3-*tert*-butyl-5-[(*Z*)-2-*tert*-butyl-4-methyl-Δ^2-1,3,4-thiadiazolin-5-ylidene]-1-formazano]-4-methyl-Δ^2-1,3,4-thiadiazolin-5-one, $M_r = 536{\cdot}71$, monoclinic, $P2_1/c$, $a = 17{\cdot}931$ (3), $b = 13{\cdot}311$ (3), $c = 26{\cdot}076$ (5) Å, $\beta = 102{\cdot}40$ (3)°, $V = 6078$ (2) Å^3, $Z = 8$, $D_x = 1{\cdot}173$ g cm^{-3}, $F(000) = 2288$, room temperature [298 (2) K], λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 18{\cdot}2824$ cm^{-1}, 643 parameters refined, final $R = 0{\cdot}094$ for all 6259 reflections. The structure of an undesirable blue compound formed during the manufacturing process of the herbicide metribuzin has been identified.

Introduction. The manufacture of the herbicide metribuzin (1) is periodically complicated by the formation of an undesirable blue compound of unknown structure. Because the occurrence of the blue dye could not be predicted or controlled, we initiated a program to determine its structure and how it is formed. Our study showed that the dye is formed by the oxidation of the reaction product of two impurities formed during the synthesis of (1) (Jackman, 1987), and this finding provided a semi-empirical synthesis for the dye. Since routine spectroscopic techniques (^{1}H and ^{13}C NMR, IR, UV, MS) did not unambiguously establish the structure, we have carried out an X-ray analysis of the trimethylated derivative and established its structure.

Experimental. Many attempts to obtain X-ray quality crystals of the dye and its metal salts were unsuccessful. We then tried various derivatives and finally succeeded with the trimethylated compound. A black plate, 0·4 × 0·2 × 0·1 mm, obtained by recrystallization from 2-propanol, was mounted on a glass fiber oriented approximately along [2$\bar{1}\bar{1}$]. Cell constants were determined by using 15 centered reflections widely scattered throughout reciprocal space ($35 > 2\theta > 25°$). Preliminary counter data indicated a monoclinic system with systematic absences, $k = 2n + 1$ in $0k0$ and $l = 2n + 1$ in $h0l$, uniquely determining the space group as $P2_1/c$. Four octants ($h = 0 \rightarrow 17$, $k = -13 \rightarrow 13$, $l = -25 \rightarrow 25$) of data out to $2\theta = 100°$ were collected using a Syntex $P2_1$ diffractometer (Cu $K\alpha$, graphite monochromator) with θ–2θ scan mode (scan angle:

0108-2701/88/081418-04$03.00

$0{\cdot}2823\tan\theta + 2{\cdot}8°$, scan speed: 1·0 to 20·0° min^{-1}, background: 1/6 of total scan at both edges of the peak). The total number of reflections measured was 12 830. Lorentz and polarization factors were applied. Correction was made for a 19% decline in intensity of two reflections monitored every 100 measurements. A semi-empirical ψ-scan technique was used to correct for absorption (±4·3%) (North, Phillips & Mathews, 1968). 6259 independent F_o^2 data were obtained by merging equivalent reflections. R_{int} for merging was 0·079. The reflections with $F_o^2 < 0{\cdot}2\sigma(F_o^2)$ were reset to $F_o^2 = 0{\cdot}2\sigma(F_o^2)$. The $\sigma(F_o^2)$ and $\sigma(F_o)$ values were defined as follows: $\sigma(F_o^2) = [\sigma_{count}^2 + (0{\cdot}02F_o^2)^2]^{1/2}$ and $\sigma(F_o) = \sigma(F_o^2)/2F_o$. All reflections were used in subsequent calculations.

Initial attempts to solve the structure automatically using the program *MULTAN*78 (Main, Hull, Lessinger, Germain, Delcercq & Woolfson, 1978) were not successful. Instead, a symbolic addition technique was used. Three origin reflections were selected; seven additional reflections were assigned symbols. After seven cycles of the symbolic addition procedure, several intersymbol relations reduced the number of independent symbols to three. After an additional five cycles, the E map with the lowest $R_{Karle} = 0{\cdot}24$ showed two thiadiazole rings and several other chemically reasonable fragments. Successive difference Fourier syntheses revealed the positions of 66 atoms in the two independent molecules. The *tert*-butyl groups, $-C(CH_3)_3$, attached to one of the thiadiazole rings in both molecules, were found to be approximately twofold disordered along the C(31)—C(33) bonds. The occupancy factors for those methyl carbons were set to 0·5. No H atoms were located from difference maps. A block-diagonal-matrix least-squares procedure was used to refine the atomic parameters, including anisotropic thermal parameters, of all non-hydrogen atoms, except for disordered methyl carbons (isotropic). The scale and overall thermal parameters were placed in a (2 × 2) matrix; the parameters of each disordered methyl carbon were placed in a (4 × 4) matrix; and the positional and thermal parameters of each remaining atom were placed in a (9 × 9) matrix. Function minimized was: $\sum w(|F_o| - |F_c|)^2$ with $w = 1/\sigma(F_o)^2$. Refinement converged to $R = 0{\cdot}094$, $wR = 0{\cdot}103$, $S = 1{\cdot}211$ for all data (6259 reflections). The maximum Δ/σ in the last block-diagonal least-squares refinement cycle is 0·35. The final difference Fourier map was featureless (±0·33 e $Å^{-3}$). Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed on a Honeywell 66/6000 computer at the University of Kansas using programs of the *KUDNA* system (Takusagawa, 1984).

Discussion. The structure of the trimethylated derivative (2) of an undesirable blue compound (3)

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters*

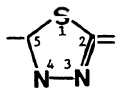
The thermal parameters are of the form $B_{eq} = \frac{8}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq} (Å²)
C(1)	0·6893 (5)	0·0767 (5)	0·9220 (4)	7·46
O(2)	0·6479 (2)	0·1705 (3)	0·9183 (2)	5·54
C(3)	0·6624 (4)	0·2380 (5)	0·8876 (2)	5·16
O(4)	0·7175 (3)	0·2384 (4)	0·8688 (3)	7·41
C(5)	0·6075 (3)	0·3290 (4)	0·8803 (1)	3·57
C(6)	0·6322 (3)	0·4184 (4)	0·9173 (2)	4·42
C(7)	0·5716 (4)	0·5028 (5)	0·9007 (4)	6·92
C(8)	0·6320 (9)	0·3805 (6)	0·9743 (3)	9·65
C(9)	0·7044 (4)	0·4530 (7)	0·9118 (5)	9·31
N(10)	0·5477 (2)	0·3246 (3)	0·8458 (2)	4·30
N(11)	0·5407 (3)	0·2302 (3)	0·8194 (2)	4·93
C(12)	0·4788 (3)	0·2255 (4)	0·7828 (2)	4·04
N(13)	0·4582 (2)	0·1390 (3)	0·7557 (2)	3·90
C(14)	0·5043 (3)	0·0525 (3)	0·7606 (2)	4·84
N(15)	0·3958 (2)	0·1441 (3)	0·7149 (2)	3·66
C(16)	0·3627 (3)	0·2304 (3)	0·7141 (2)	3·65
S(17)	0·40813 (6)	0·31853 (7)	0·76150 (4)	3·64
N(18)	0·2990 (2)	0·2486 (3)	0·6773 (2)	3·76
N(19)	0·2749 (2)	0·3385 (3)	0·6794 (1)	3·46
C(20)	0·2107 (2)	0·3591 (3)	0·6385 (2)	2·86
C(21)	0·1754 (3)	0·2935 (4)	0·5937 (2)	4·18
C(22)	0·1478 (3)	0·1920 (4)	0·6141 (3)	5·59
C(23)	0·1103 (5)	0·3474 (7)	0·5537 (4)	9·24
C(24)	0·2352 (5)	0·2626 (7)	0·5634 (3)	7·86
N(25)	0·1794 (2)	0·4517 (3)	0·6396 (2)	3·54
N(26)	0·2088 (2)	0·5127 (3)	0·6808 (2)	3·61
C(27)	0·1714 (3)	0·5978 (3)	0·6728 (2)	3·76
N(28)	0·1870 (3)	0·6733 (4)	0·7062 (1)	4·20
C(29)	0·2460 (4)	0·6766 (4)	0·7535 (2)	6·16
N(30)	0·1449 (3)	0·7616 (3)	0·6900 (2)	4·69
C(31)	0·0989 (3)	0·7519 (3)	0·6484 (2)	3·30
S(32)	0·09785 (7)	0·63092 (9)	0·62029 (5)	4·22
C(33)	0·0435 (3)	0·8317 (4)	0·6191 (2)	4·34
C(34)*	0·033 (1)	0·826 (1)	0·6374 (8)	6·99
C(35)*	0·029 (1)	0·812 (1)	0·5596 (8)	11·23
C(36)*	0·079 (1)	0·937 (1)	0·6309 (8)	9·26
C(37)*	0·021 (1)	0·905 (1)	0·6589 (8)	9·82
C(38)*	−0·029 (1)	0·780 (1)	0·5877 (8)	12·31
C(39)*	0·083 (1)	0·890 (1)	0·5812 (8)	6·88
C(1′)	−0·2014 (7)	0·568 (1)	0·5799 (4)	11·18
O(2′)	−0·1516 (3)	0·4845 (3)	0·5830 (2)	6·01
C(3′)	−0·1674 (3)	0·4157 (4)	0·6138 (2)	4·14
O(4′)	−0·2191 (3)	0·4174 (4)	0·6349 (3)	7·46
C(5′)	−0·1112 (2)	0·3255 (4)	0·6171 (2)	4·24
C(6′)	−0·1327 (2)	0·2381 (4)	0·5849 (2)	3·74
C(7′)	−0·0713 (4)	0·1519 (7)	0·5930 (3)	7·34
C(8′)	−0·1541 (9)	0·2711 (7)	0·5315 (4)	10·29
C(9′)	−0·2114 (6)	0·1883 (8)	0·5995 (6)	10·72
N(10′)	−0·0472 (2)	0·3324 (3)	0·6525 (2)	3·99
N(11′)	−0·0389 (3)	0·4271 (3)	0·6788 (2)	4·61
C(12′)	0·0239 (2)	0·4295 (3)	0·7132 (2)	3·05
N(13′)	0·0424 (2)	0·5139 (3)	0·7454 (1)	3·53
C(14′)	−0·0066 (3)	0·6086 (4)	0·7367 (2)	4·93
N(15′)	0·1046 (2)	0·5113 (2)	0·7807 (1)	3·29
C(16′)	0·1381 (3)	0·4248 (3)	0·7804 (2)	3·47
S(17′)	0·09281 (6)	0·33728 (8)	0·73512 (4)	3·59
N(18′)	0·2041 (2)	0·4049 (3)	0·8206 (2)	3·42
N(19′)	0·2278 (2)	0·3176 (2)	0·8172 (2)	3·45
C(20′)	0·2920 (4)	0·2932 (4)	0·8558 (2)	5·09
C(21′)	0·3225 (3)	0·3650 (3)	0·9027 (2)	3·86
C(22′)	0·3868 (3)	0·3064 (5)	0·9406 (2)	5·47
C(23′)	0·3539 (3)	0·4564 (4)	0·8856 (2)	4·20
C(24′)	0·2572 (4)	0·3885 (4)	0·9344 (2)	5·11
N(25′)	0·3218 (2)	0·2048 (3)	0·8563 (2)	3·78
N(26′)	0·2953 (2)	0·1435 (3)	0·8142 (1)	3·52
C(27′)	0·3329 (2)	0·0559 (3)	0·8247 (1)	2·83
N(28′)	0·3205 (3)	−0·0238 (3)	0·7924 (2)	4·24
C(29′)	0·2542 (5)	−0·0292 (4)	0·7458 (3)	7·44
N(30′)	0·3607 (2)	−0·1092 (3)	0·8100 (2)	4·25
C(31′)	0·4059 (3)	−0·0941 (3)	0·8522 (2)	4·02
S(32′)	0·40303 (7)	0·02799 (8)	0·87951 (5)	4·03
C(33′)	0·4545 (3)	−0·1767 (4)	0·8783 (3)	4·95
C(34′)*	0·413 (1)	−0·233 (1)	0·9154 (8)	11·58
C(35′)*	0·530 (1)	−0·133 (1)	0·9102 (8)	11·18
C(36′)*	0·472 (1)	−0·250 (1)	0·8366 (8)	11·55
C(37′)*	0·413 (1)	−0·278 (1)	0·8647 (8)	8·18
C(38′)*	0·471 (1)	−0·161 (1)	0·9382 (8)	9·18
C(39′)*	0·530 (1)	−0·178 (1)	0·8594 (8)	10·50

* The occupancy factors of atoms with * are 0·5.

Table 2. *Bond distances* (Å) *and angles* (°) *of thiadiazole rings found in this study and in the Cambridge Structural Database*

The atom numbering is shown below.



Code*	R	S_1–C_2	C_2=N_3	N_3–N_4	N_4–C_5	C_5–S_1	$\angle S_1$	$\angle C_2$	$\angle N_3$	$\angle N_4$	$\angle C_5$
This study	0·094	1·771	1·291	1·370	1·360	1·774	86·94	115·93	110·64	116·55	109·41
This study	0·094	1·732	1·299	1·285	1·399	1·748	86·88	116·77	110·53	117·75	107·92
This study	0·094	1·768	1·220	1·412	1·320	1·742	88·04	114·20	113·19	114·37	110·09
This study	0·094	1·779	1·235	1·371	1·343	1·729	86·78	115·81	111·13	116·14	109·98
BIPTDZ	0·088	1·742	1·305	1·384	1·351	1·755	88·49	115·71	109·57	117·26	108·97
CIDSOX	0·053	1·714	1·293	1·363	1·371	1·742	86·97	119·26	107·29	117·43	109·02
DMITDZ	0·052	1·740	1·288	1·383	1·354	1·720	87·75	117·73	107·27	117·84	109·38
MIMTBH	0·067	1·736	1·276	1·375	1·329	1·710	86·26	119·07	105·97	118·25	110·45
MPTZTD	0·082	1·758	1·302	1·364	1·373	1·740	88·36	116·41	108·24	118·99	107·98
MSNROD	0·188	1·758	1·333	1·371	1·293	1·722	87·66	108·15	118·91	107·75	117·49
MSTAZO	0·160	1·744	1·351	1·383	1·304	1·727	89·04	108·78	116·96	109·65	115·57

* Reference codes used in the Cambridge Structural Database are:
BIPTDZ: 2-benzoylimino-3,5-diphenyl-2,3-dihydro-1,3,4-thiadiazole (Fukutani, Tsukihara, Okuda, Fukuyama, Katsube, Yamamoto & Gotoh, 1979).
CIDSOX: 2-ethylsulfonyl-7-methyl-5*H*-1,3,4-thiadiazolo[3,2-*a*]pyrimidin-5-one (Suiko, Nakatsu, Kiyose & Imada, 1984).
DMITDZ: 5,6-dimethyl-imidazo[2,1-*b*][1,3,4]thiadiazole (Schenetti *et al.*, 1982).
MIMTBH: 5,6-dimethyl-imidazo[2,1-*b*][1,3,4]thiadiazole hydrobromide monohydrate (Schenetti *et al.*, 1982).
MPTZTD: 2-methyl-5-phenyl-*S*-triazolo[3,4-*b*]-1,3,4-thiadiazole (Fornies-Marquina, Courseille & Elguero, 1974).
MSNROD: 5-(2-methylmercapto-4-methyl-4,5-dihydro-1′,3′,4′-thiadiazol-5-ylidene)-3-ethyl-rhodanine-iodine (Bois D'Enghien-Peteau, Meunier-Piret & Van Meerssche, 1968).
MSTAZO: 3-methyl-5-thiomethyl-2,3-dihydro-1,3,4-thiadiazole-2-(1′-ethyl-4′-thiazolidinylidene-2′-thione-5′-one) (Germain, Paternotte, Piret & Van Meerssche, 1964).

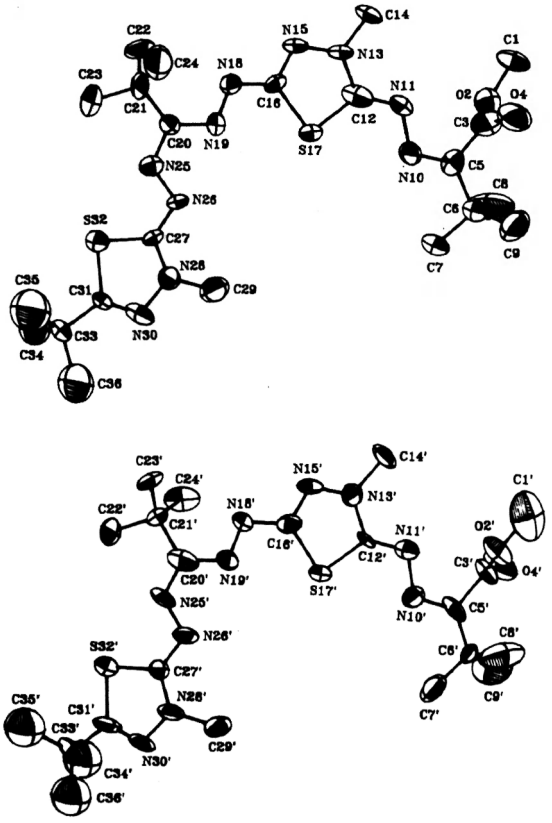


Fig. 1. View of the title compound (2) with atom numbering. The disordered C(37), C(38) and C(39) atoms are omitted in the figures.

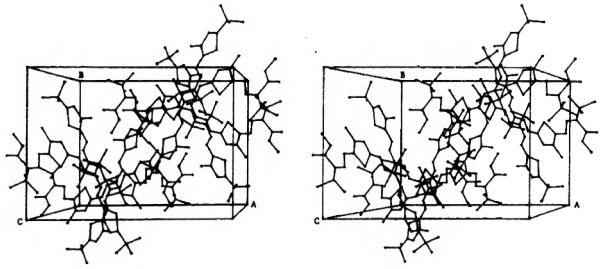
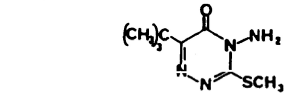


Fig. 2. Stereoscopic drawing showing the crystal structure viewed approximately along the *c* axis.

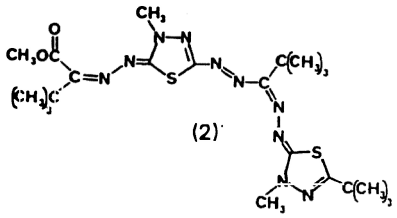
formed in the process of manufacture of the herbicide metribuzin (1) has been determined. Final fractional coordinates with equivalent isotropic temperature factors are listed in Table 1. Bond distances and angles are listed in Tables 4 and 5.* Two crystallographically independent molecules are identical to each other as shown in Fig. 1. The molecules are essentially flat except for the terminal groups as shown in Table 7.* These two independent molecules are related to each other by a pseudo-center of symmetry at (0·24961,

* Lists of complete molecular dimensions [bond distances (Table 4), angles (Table 5), torsion angles (Table 6) and least-squares-planes calculations (Table 7)], anisotropic thermal parameters (Table 3), structure factors (Table 8) and the preparation process of (2) from (3) and the physical data of (2) and (3) (Table 9) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44914 (58 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

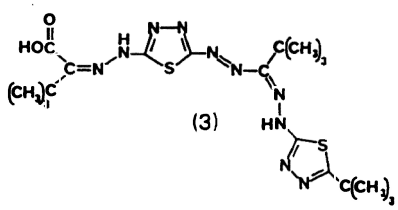
0·32694, 0·74936) as shown in Fig. 2. The maximum deviation from the pseudo-center is 0·213 Å for the pair of C(8) and C(8′), and the mean deviation is 0·060 Å. The dimensions of the rather rare thiadiazole rings found in this study are listed in Table 2 along with those found in the Cambridge Structural Database (Allen *et al.*, 1979). Structure (2), determined by this X-ray analysis, and the NMR and MS data suggest structure (3) for the dye.



(1)



(2)



(3)

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Structure of a New Clerodane Derivative from *Tinospora cordifolia* Miers

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Abstract. (5*R*,10*R*)-4*R*,8*R*-Dihydroxy-2*S*,3*R*:15,16-diepoxycleroda-13(16),14-dieno-17,12*S*:18,1*S*-dilactone, $C_{20}H_{22}O_8$, $M_r = 390{\cdot}20$, m.p. = 504–506 K, monoclinic, $P2_1$, $a = 7{\cdot}969\ (3)$, $b = 12{\cdot}833\ (5)$, $c = 8{\cdot}627\ (2)$ Å, $\beta = 101{\cdot}60\ (2)°$, $V = 864{\cdot}11$ Å^3, $Z = 2$, $D_m = 1{\cdot}490$ (by flotation in $CCl_4 + CHCl_3$), $D_x = 1{\cdot}498$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 8{\cdot}77$ cm^{-1}, $F(000) = 412$, $T = 295$ K, $R = 0{\cdot}0367$, $wR = 0{\cdot}0445$ for 1654 observed reflections. The structure contains two terpene rings, an epoxy group, two δ-lactones, two tertiary hydroxyl groups, two methyl groups, and a β-substituted furan moiety. The terpene ring *A* is locked into a boat conformation by the C(1)–C(4) lactone bridge. The furan ring is attached equatorially to atom C(12). The structure is stabilized by a network of hydrogen bonds involving the two hydroxyl groups.

Introduction. *Tinospora cordifolia* Miers belongs to the family Menispermaceae. It has been shown to possess several pharmacological activities (Nirmala, Sharadini

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