

plane is 0.074 Å. The considerable distortions in the central part of the Thy[1(CH₂—CHOH—CH₂)1']Thy molecule are due to stresses caused by the interactions of the terminal substituents (Table 3). The presence of the hydroxyl group in the molecule of Thy[1(CH₂—CHOH—CH₂)1']Thy causes the packing mode to differ from that of Thy[1(CH₂)₃1']Thy. The hydroxyl O(8) is involved with the carbonyl O(2) in close intramolecular contact and with the carbonyl O(4') in the intermolecular hydrogen bond. The protonated N(3) and N(3') are hydrogen bonded to the carbonyl O(2) and O(2'), respectively. In this way thymine bases are connected in pairs about two kinds of symmetry centers.

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Structure of 3',8-Bis[*p*-chlorophenylthiomethoxy]-4',5,6',7-tetramethylchroman-2-spiro-1'-cyclohexa-3',5'-dien-2'-one: a Spirocyclic Chroman

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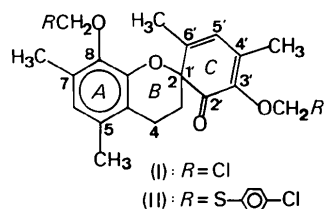
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(Received 7 August 1981; accepted 1 October 1981)

Abstract. C₃₂H₃₀Cl₂O₄S₂, triclinic, $P\bar{1}$, $a = 7.636$ (5), $b = 14.167$ (7), $c = 15.186$ (8) Å, $\alpha = 108.75$ (6), $\beta = 100.54$ (6), $\gamma = 77.41$ (6)°, $Z = 2$, $F(000) = 640$, $\mu(\text{Cu } K\alpha) = 3.39$ mm⁻¹. Final $R = 0.065$ for 2534 observed reflections. The conformation and dimensions of the pyranspirocyclohexadienone moiety are similar to those observed in the 3',8-bis(chloromethoxy) analogue [Cacioli, Mackay & Reiss (1980). *Tetrahedron Lett.* **21**, 4973–4976; Cacioli, Mackay & Reiss (1981). *Acta Cryst.* **B37**, 1620–1622]. The pyran ring system is in a half-chair form and the cyclohexadienone ring is envelope. In the 1,3-diene moiety, the two long and two short C—C bonds have mean values of 1.461 (8) and 1.332 (8) Å respectively.

Introduction. During the course of an investigation of the chemistry of spiroepoxycyclohexadienones, the spirocyclic chroman (I) was obtained (Cacioli *et al.*,

1980, 1981). Although spirocyclic chromans are known to undergo a number of acid-promoted rearrangement reactions (Dick, Dean, Matkin & Robinson, 1977), there are few reports of their reactions with nucleophiles. Reactions between chroman (I) and sodium methoxide, or ethoxide, gave complex mixtures of products (Cacioli & Reiss, 1981). However, on reaction with sodium *p*-chlorothiophenoxide in methanol, chroman (I) gave the nucleophilic displacement product (II). Because of ambiguity in the ¹H and ¹³C spectral data of compound (II) by comparison with that of compound (I) (Cacioli & Reiss, 1981), an X-ray structure determination of (II) was carried out.



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Table 1. Final atomic parameters of the non-H atoms ($\times 10^4$) and equivalent isotropic temperature factors with e.s.d.'s in parentheses

	$B_{\text{eq}} = 8\pi^2 U_{\text{eq}}$, where $U_{\text{eq}} = (U_1 U_2 U_3)^{1/3}$.			
	x	y	z	$B_{\text{eq}} (\text{\AA}^2)$
Cl(1)	9634 (3)	2894 (2)	8270 (1)	6.7 (1)
Cl(2)	1158 (3)	747 (2)	-6080 (1)	7.1 (1)
S(1)	4818 (3)	3715 (1)	4688 (1)	4.4 (1)
S(2)	5198 (3)	1967 (2)	-2013 (1)	6.3 (1)
O(1)	4733 (5)	2351 (3)	1711 (2)	3.3 (2)
O(2)	6740 (7)	1411 (3)	258 (3)	3.0 (2)
O(3)	6823 (6)	2410 (3)	3347 (2)	3.7 (2)
O(4)	5260 (7)	285 (3)	-1519 (3)	4.8 (3)
C(1)	5996 (9)	3700 (4)	1505 (4)	3.6 (3)
C(2)	5867 (8)	3058 (4)	2009 (4)	3.0 (3)
C(3)	6913 (9)	3099 (4)	2877 (4)	3.3 (3)
C(4)	8140 (9)	3760 (5)	3246 (4)	3.8 (3)
C(5)	8263 (9)	4394 (5)	2726 (4)	4.3 (4)
C(6)	7239 (10)	4379 (5)	1881 (4)	4.4 (4)
C(7)	4856 (10)	3650 (5)	585 (4)	4.1 (4)
C(8)	3223 (10)	3142 (4)	482 (4)	4.1 (3)
C(9)	3829 (10)	2137 (4)	768 (3)	3.0 (4)
C(10)	2154 (10)	1731 (5)	745 (4)	3.9 (3)
C(11)	1544 (9)	1084 (5)	-48 (4)	4.0 (4)
C(12)	2556 (11)	648 (4)	-849 (4)	3.4 (3)
C(13)	4226 (11)	817 (4)	-793 (4)	3.3 (3)
C(14)	5092 (11)	1427 (4)	90 (4)	3.3 (3)
C(15)	5178 (10)	2573 (5)	3728 (4)	4.3 (4)
C(16)	9329 (10)	3775 (5)	4153 (4)	4.8 (4)
C(17)	7450 (12)	5077 (5)	1343 (5)	6.2 (5)
C(18)	1126 (10)	2171 (6)	1588 (5)	5.8 (4)
C(19)	1680 (10)	-39 (5)	-1736 (4)	4.7 (4)
C(20)	6394 (11)	826 (8)	-1773 (5)	5.7 (5)
C(21)	6295 (8)	3450 (4)	5650 (4)	3.5 (3)
C(22)	7565 (9)	2570 (5)	5605 (4)	4.7 (4)
C(23)	8582 (10)	2389 (5)	6403 (5)	4.9 (4)
C(24)	8378 (9)	3128 (6)	7252 (4)	4.2 (4)
C(25)	7192 (10)	4021 (5)	7314 (4)	4.8 (4)
C(26)	6174 (9)	4178 (5)	6514 (4)	4.4 (3)
C(27)	4083 (9)	1551 (6)	-3161 (4)	4.2 (4)
C(28)	3731 (11)	572 (6)	-3588 (4)	5.8 (4)
C(29)	2860 (11)	322 (6)	-4500 (5)	5.9 (4)
C(30)	2311 (10)	1046 (6)	-4951 (4)	4.9 (4)
C(31)	2634 (11)	2008 (7)	-4529 (5)	5.8 (5)
C(32)	3507 (11)	2249 (6)	-3633 (5)	5.7 (5)

Weissenberg photographs showed that the yellow tabular crystals grown from diethyl ether were triclinic. Intensities were measured with Cu $K\alpha$ radiation (graphite-crystal monochromator, $\lambda = 1.5418 \text{ \AA}$) from a crystal $ca\ 0.10 \times 0.18 \times 0.14 \text{ mm}$ aligned with **b** approximately parallel to the φ axis of a Rigaku-AFC four-circle diffractometer. Of the 4977 non-equivalent terms ($2\theta_{\text{max}} = 129^\circ$) recorded by an $\omega-2\theta$ scan of 2° min^{-1} in 2θ and with 10 s stationary background counts, the 2534 for which $|F_o| > 3\sigma|F_o|$ were used for the structure refinement. The intensities were corrected for Lorentz and polarization effects and for absorption. The scattering factors for Cl, S, O and C were from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965). Anomalous-dispersion corrections were made for the non-H atoms (Cromer & Liberman, 1970).

The structure was solved by direct methods with *SHELX 76* (Sheldrick, 1976). Full-matrix least-squares refinement with anisotropic temperature factors given to the non-H atoms converged to give $R = 0.065$ and $R_w = (\sum w||F_o| - |F_c||^2 / \sum w|F_o|^2)^{1/2} = 0.069$. Although maxima at the expected H-atom sites were noted on the difference maps, not all were clearly

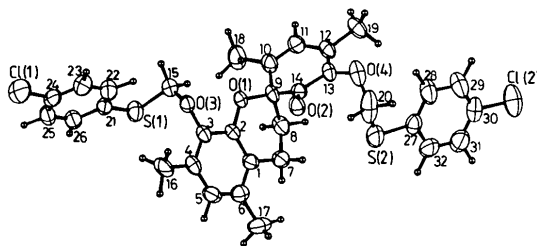


Fig. 1. Perspective view of the molecule with thermal ellipsoids scaled to 50% probability. The H atoms are represented by spheres of arbitrary radius. The C atoms are denoted by numerals only.

Table 2. Selected torsional angles ($^\circ$)

E.s.d.'s range from 0.6 to 0.8 $^\circ$. Values for the 3',8-bis(chloromethoxy) analogue (Cacioli *et al.*, 1981) are included in parentheses.

C(1)-C(2)-O(1)-C(9)	-10.2	(-9.9)	C(14)-C(9)-C(10)-C(11)	27.8	(26.0)
C(2)-O(1)-C(9)-C(8)	38.4	(35.1)	C(2)-O(1)-C(9)-C(10)	155.7	(152.4)
O(1)-C(9)-C(8)-C(7)	-57.0	(-54.7)	C(2)-O(1)-C(9)-C(14)	-79.9	(-83.1)
C(9)-C(8)-C(7)-C(1)	47.3	(48.2)	C(7)-C(8)-C(9)-C(10)	-174.4	(-173.3)
C(8)-C(7)-C(1)-C(2)	-19.9	(-23.5)	C(7)-C(8)-C(9)-C(14)	63.2	(64.6)
C(7)-C(1)-C(2)-O(1)	-0.3	(3.4)	C(11)-C(10)-C(9)-C(8)	-90.9	(-92.1)
O(1)-C(2)-C(3)-O(3)	-3.4	(-6.3)	C(11)-C(10)-C(9)-O(1)	150.6	(148.0)
C(2)-C(3)-O(3)-C(15)	71.1	(69.9)	C(13)-C(14)-C(9)-O(1)	-155.2	(-153.6)
O(3)-C(3)-C(4)-C(16)	2.0	(2.5)	C(13)-C(14)-C(9)-C(8)	85.1	(86.0)
C(3)-O(3)-C(15)-S(1)	67.9		C(12)-C(13)-O(4)-C(20)	-141.4	(-140.5)
O(3)-C(15)-S(1)-C(21)	74.5		C(14)-C(13)-O(4)-C(20)	48.8	(50.8)
C(15)-S(1)-C(21)-C(22)	-5.9		C(13)-O(4)-C(20)-S(2)	53.1	
			O(4)-C(20)-S(2)-C(27)	78.8	
C(7)-C(1)-C(6)-C(17)	0.4	(-0.5)	C(20)-S(2)-C(27)-C(28)	-20.6	
C(9)-C(10)-C(11)-C(12)	-8.6	(-7.9)	O(4)-C(13)-C(14)-O(2)	7.7	(6.4)
C(10)-C(11)-C(12)-C(13)	-5.6	(-5.8)	O(2)-C(14)-C(9)-C(8)	-91.1	(-91.9)
C(11)-C(12)-C(13)-C(14)	-1.6	(-1.1)	O(1)-C(9)-C(10)-C(18)	-36.4	(-37.8)
C(12)-C(13)-C(14)-C(9)	21.9	(20.3)	O(2)-C(14)-C(9)-O(1)	28.6	(28.6)
C(13)-C(14)-C(9)-C(10)	-33.8	(-31.9)	O(4)-C(13)-C(12)-C(19)	6.3	(7.6)

resolved. Consequently, the H atoms were included with calculated coordinates and were given a common isotropic temperature factor which refined to a value $B = 10.7 (5) \text{ \AA}^2$. The function minimized in the least-squares refinements was $\sum w(|F_o| - |F_c|)^2$ with the terms weighted according to $\sigma^2(|F_o| + 0.0005|F_c|)^{-1}$. The maximum parameter shift-to-error ratio at convergence was 0.05 : 1, and the largest peaks on the final difference map were of heights 0.32 and $-0.47 e \text{ \AA}^{-3}$. Final atomic coordinates are given in Table 1.* Figs. 1 and 2 have been prepared with ORTEP (Johnson, 1965).

Discussion. The molecular structure† is illustrated in Fig. 1. The pentacyclic molecule adopts an extended conformation with the angles between the perpendiculars to rings *A* and *D* [C(27)–C(32)], *A* and *E* [C(22)–C(26)] and *D* and *E* 29.5 (5), 68.7 (7) and 39.3 (8)° respectively. The conformation of the pyran-spirocyclohexadienone moiety is similar to its conformation in (I), the chloromethoxy analogue (Cacioli *et al.*, 1980, 1981) (see torsional angles given in Table 2). Ring *B* is in the half-chair form with C(9) +0.229 (7) and C(8) –0.490 (7) Å from the plane of the other four atoms which are coplanar within ±0.001 Å. Ring *C* is envelope; C(9) is +0.388 (6) Å from the plane of the other ring atoms which form a plane with an r.m.s. deviation of 0.033 Å. The exocyclic ketonic and ether oxygen atoms, O(2) and O(4), deviate from the latter by –0.336 (4) and –0.106 (4) Å respectively so that the torsional angle O(4)–C(13)–C(14)–O(2) is 7.7° [*cf.* value 6.4° in (I)]. The torsional angles C(1)–C(2)–O(1)–C(9) –10.2 and C(8)–C(7)–C(1)–C(2) –19.9° are similar to the values –9.9 and –23.5° observed for these angles in (I). Space-filling molecular models of the latter and (II) suggest that free rotation of the chloromethyl or *p*-chlorophenylthiomethyl groups about the C(3)–O(3) and C(13)–O(4) bonds is not possible because of steric repulsion of the adjacent substituents. The torsional angle C(2)–C(3)–O(3)–C(15) which differs by only 0.9° in the two molecules, and the torsional angle C(14)–C(13)–O(4)–C(20) which differs by only 2.0°, lend support to this.

No unusual bond lengths or angles are observed (see Table 3). As expected, dimensions of ring *C* from C(10) through to C(14) indicate the presence of some conjugation. The mean values for the two shorter and

two longer C–C bonds of 1.332 (8) and 1.461 (8) Å respectively are in reasonable agreement with the values for C–C double and C(*sp*²)–C(*sp*²) single bonds reported by Lide (1962) and Stoicheff (1962). The S–C(*sp*³) bonds have a mean length 1.796 (9) Å in accord with the value 1.804 (2) Å reported for this bond in tetrakis(methylthio)ethylene (Collins & Davis, 1978*a*), while the S–Ph bonds have a mean length 1.766 (6) Å which is very similar to the values 1.767 (2) and 1.770 (2) Å in tetrakis(phenylthio)ethylene (Collins & Davis, 1978*b*). The angles subtended at the S atoms, with mean value 104.5 (4)°, can

Table 3. Bond lengths (Å) and angles (°) with *e.s.d.*'s in parentheses

Cl(1)–C(24)	1.752 (7)	C(7)–C(8)	1.531 (11)
Cl(2)–C(30)	1.743 (6)	C(8)–C(9)	1.571 (8)
S(1)–C(15)	1.804 (7)	C(9)–C(10)	1.503 (11)
S(1)–C(21)	1.764 (6)	C(9)–C(14)	1.509 (9)
S(2)–C(20)	1.787 (11)	C(10)–C(11)	1.334 (9)
S(2)–C(27)	1.768 (6)	C(10)–C(18)	1.512 (10)
O(1)–C(2)	1.379 (7)	C(11)–C(12)	1.453 (9)
O(1)–C(9)	1.439 (6)	C(12)–C(13)	1.331 (12)
O(2)–C(14)	1.234 (10)	C(12)–C(19)	1.523 (9)
O(3)–C(3)	1.401 (7)	C(13)–C(14)	1.470 (9)
O(3)–C(15)	1.421 (9)	C(21)–C(22)	1.393 (9)
O(4)–C(13)	1.390 (8)	C(21)–C(26)	1.388 (8)
O(4)–C(20)	1.445 (11)	C(22)–C(23)	1.381 (10)
C(1)–C(2)	1.392 (8)	C(23)–C(24)	1.385 (10)
C(1)–C(6)	1.414 (10)	C(24)–C(25)	1.373 (11)
C(1)–C(7)	1.494 (9)	C(25)–C(26)	1.372 (9)
C(2)–C(3)	1.402 (8)	C(27)–C(28)	1.392 (11)
C(3)–C(4)	1.386 (10)	C(27)–C(32)	1.355 (11)
C(4)–C(5)	1.400 (10)	C(28)–C(29)	1.392 (10)
C(4)–C(16)	1.501 (9)	C(29)–C(30)	1.362 (11)
C(5)–C(6)	1.372 (9)	C(30)–C(31)	1.362 (12)
C(6)–C(17)	1.519 (10)	C(31)–C(32)	1.372 (10)
C(2)–C(1)–C(6)	118.3 (6)	C(12)–C(13)–O(4)	119.6 (6)
C(2)–C(1)–C(7)	120.0 (6)	O(4)–C(13)–C(14)	119.2 (6)
C(6)–C(1)–C(7)	121.7 (6)	C(9)–C(14)–C(13)	115.9 (6)
C(1)–C(2)–O(1)	123.9 (5)	O(2)–C(14)–C(9)	121.0 (6)
C(1)–C(2)–C(3)	120.9 (6)	O(2)–C(14)–C(13)	123.0 (6)
O(1)–C(2)–C(3)	115.2 (5)	O(3)–C(15)–S(1)	114.4 (5)
C(2)–C(3)–C(4)	121.0 (6)	O(4)–C(20)–S(2)	113.4 (6)
O(3)–C(3)–C(2)	119.6 (5)	S(1)–C(21)–C(22)	125.0 (5)
O(3)–C(3)–C(4)	119.2 (6)	S(1)–C(21)–C(26)	117.1 (5)
C(3)–C(4)–C(5)	117.3 (6)	C(22)–C(21)–C(26)	118.0 (6)
C(3)–C(4)–C(16)	121.4 (6)	C(21)–C(22)–C(23)	121.3 (6)
C(5)–C(4)–C(16)	121.2 (6)	C(22)–C(23)–C(24)	118.4 (7)
C(4)–C(5)–C(6)	122.9 (6)	Cl(1)–C(24)–C(23)	118.6 (6)
C(1)–C(6)–C(5)	119.6 (6)	Cl(1)–C(24)–C(25)	119.7 (5)
C(1)–C(6)–C(17)	119.9 (6)	C(23)–C(24)–C(25)	121.7 (7)
C(5)–C(6)–C(17)	120.4 (6)	C(24)–C(25)–C(26)	119.0 (6)
C(1)–C(7)–C(8)	111.5 (5)	C(21)–C(26)–C(25)	121.6 (6)
C(7)–C(8)–C(9)	110.3 (5)	S(2)–C(27)–C(28)	124.2 (6)
C(8)–C(9)–O(1)	109.7 (5)	S(2)–C(27)–C(32)	116.8 (6)
C(8)–C(9)–C(14)	107.4 (5)	C(28)–C(27)–C(32)	119.0 (7)
C(8)–C(9)–C(10)	107.7 (5)	C(27)–C(28)–C(29)	119.4 (7)
C(10)–C(9)–O(1)	108.1 (5)	C(28)–C(29)–C(30)	119.6 (7)
C(10)–C(9)–C(14)	113.3 (5)	Cl(2)–C(30)–C(29)	119.8 (6)
O(1)–C(9)–C(14)	110.5 (5)	Cl(2)–C(30)–C(31)	119.2 (6)
C(9)–C(10)–C(11)	118.7 (6)	C(29)–C(30)–C(31)	121.0 (7)
C(9)–C(10)–C(18)	117.9 (6)	C(30)–C(31)–C(32)	119.2 (8)
C(11)–C(10)–C(18)	123.0 (7)	C(31)–C(32)–C(27)	121.7 (8)
C(10)–C(11)–C(12)	122.8 (6)	C(15)–S(1)–C(21)	105.0 (3)
C(11)–C(12)–C(13)	120.6 (6)	C(20)–S(2)–C(27)	104.0 (4)
C(11)–C(12)–C(19)	118.1 (6)	C(2)–O(1)–C(9)	119.2 (4)
C(13)–C(12)–C(19)	121.2 (6)	C(3)–O(3)–C(15)	114.9 (5)
C(12)–C(13)–C(14)	120.4 (6)	C(13)–O(4)–C(20)	117.6 (6)

* Lists of structure amplitudes, calculated H-atom coordinates, anisotropic thermal parameters and least-squares planes with atom deviations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36450 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† A different numbering for the atoms is used from that for the systematic nomenclature given in (II).

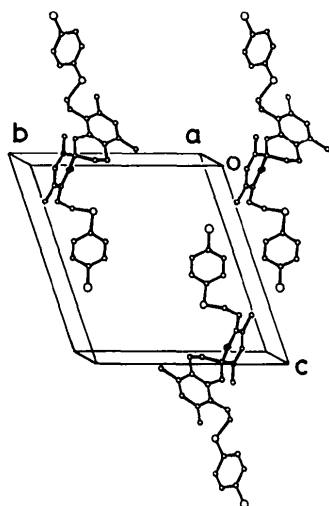


Fig. 2. The crystal packing.

Table 4. Intramolecular approaches less than 3.70 Å

E.s.d.'s are 0.01 Å. Transformations of the coordinates x, y, z are denoted by superscripts: (i) $1-x, 1-y, 1-z$; (ii) $-x, -y, -1-z$; (iii) $1-x, -y, -z$; (iv) $1+x, y, z$; (v) $x, y, 1+z$; (vi) $1+x, y, 1+z$; (vii) $1-x, y, -1-z$.

S(1)···S(1 ⁱ)	3.51	C(5)···C(18 ^{iv})	3.55
Cl(2)···C(19 ⁱⁱ)	3.59	C(12)···C(14 ⁱⁱⁱ)	3.51
O(1)···O(4 ⁱⁱⁱ)	3.65	C(13)···C(14 ⁱⁱⁱ)	3.57
O(2)···C(11 ^{iv})	3.42	C(21)···C(32 ^v)	3.50
O(2)···C(11 ^{iv})	3.69	C(22)···C(32 ^v)	3.66
O(2)···C(12 ⁱⁱⁱ)	3.23	C(23)···C(30 ^{vi})	3.64
O(2)···C(19 ⁱⁱⁱ)	3.33	C(23)···C(31 ^{vi})	3.50
O(3)···C(19 ⁱⁱⁱ)	3.56	C(26)···C(32 ^v)	3.68
O(4)···C(10 ⁱⁱⁱ)	3.51	C(29)···C(29 ^{vii})	3.67
C(4)···C(18 ^{iv})	3.58		

be compared with the values in the two thioethylene derivatives which range between 102.5 (1) and 103.6 (1)°.

The molecular packing is illustrated in Fig. 2. The molecules are aligned with their long axes along c . There are no unusually short intermolecular contacts (see Table 4). The two closest involve the oxo oxygen, O(2), which lies 3.23 Å from C(12) and 3.33 Å from C(19) of an adjacent molecule related by the inversion centre at $[\frac{1}{2}00]$.

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Acta Cryst. (1982). **B38**, 1004–1007

Conformation and Structure of *cyclo(-N-Benzylglycyl-L-prolyl-L-prolyl-)*

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(Received 5 August 1981; accepted 1 October 1981)

Abstract. C₁₉H₂₃N₃O₃, $M_r = 341.40$, orthorhombic, $P2_12_12_1$, $Z = 4$, $a = 5.688$ (2), $b = 11.782$ (3), $c = 25.727$ (9) Å, $V = 1724.0$ (9) Å³, $D_c = 1.315$ Mg m⁻³; single-crystal diffractometer data up to $\sin \theta/\lambda = 0.48$ Å⁻¹; $R_w(F) = 0.027$ for 909 independent reflections. The molecule has a crown conformation. One of

the three *cis* peptide bonds deviates significantly from planarity.

Introduction. The title compound was synthesized by Krämer (1976). Single crystals were obtained by recrystallization from chloroform. A specimen with