

Scan width 0.6° above K α ₁ and 0.6° below K α ₂, variable scan rate, background counts on each side of scan, refinement by full-matrix least-squares methods. H-atom positional parameters for compounds (I), (II) and (III) were calculated using ideal geometries and were allowed to ride on their attached atoms, C—H 0.97 and N—H 1.00 Å. Compounds (I), (II) and (III) display disorder of the H atoms attached to C2'. Compounds (I) and (III) also display disorder of the H atoms attached to C6'. Compound (I) has a disordered side chain where C18 and C19 show alternate positions of half occupancy each. Compound (II) displays disorder of C18 with an alternate position of half occupancy.

For all compounds, data collection: XSCANS (Siemens, 1991); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structures: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structures: SHELXS86 for (I); SHELXL93 (Sheldrick, 1993) for (II) and (III). For all compounds, molecular graphics: XP (Siemens, 1990)

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1223). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(E)-6-Chloro-3-[2-(4-chlorophenylsulfonyl)ethenyl]-4-chromanone, (E)-6-Bromo-3-[2-(4-bromophenylsulfonyl)ethenyl]-4-chromanone and (E)-3-[2-(4-Chlorophenylsulfonyl)ethenyl]-6-methoxy-4-chromanone

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Abstract

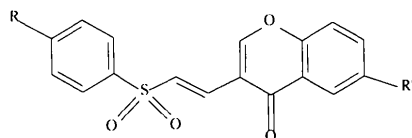
The structures of (E)-6-chloro-3-[2-(4-chlorophenylsulfonyl)ethenyl]-4-chromanone, C₁₇H₁₀Cl₂O₄S, (E)-6-bromo-3-[2-(4-bromophenylsulfonyl)ethenyl]-4-chromanone, C₁₇H₁₀Br₂O₄S, and (E)-3-[2-(4-chlorophenylsulfonyl)ethenyl]-6-methoxy-4-chromanone, C₁₈H₁₃ClO₅S, display similar bond angles and distances, but differ in the conformations of the ring systems.

Comment

Sulfones display activity as antibacterial and antifungal agents. Dapsone has been proven to be effective against leprosy, and diasone is found to be highly effective against streptococci and pneumococci infections (Khairesch, Stampa & Nudenberg, 1953). The antifungicidal activity of some unsaturated sulfones has been found to be dependent upon substituent and stereochemical effects (Hawthorne, 1960). (E)-3-[2-(Phenylsulfonyl)ethenyl]-4H-1-benzopyran-4-one and (E)-3-[2-(4-chlorophenylsulfonyl)ethenyl]-4H-1-benzopyran-4-one have been observed to display antifungal activity against *Curvularia lunata* and *Fusarium oxysporum* (Mukundam, 1990).

In the interest of exploiting and increasing this activity, we have synthesized a series of compounds which are derived from these active antifungal agents but with substituents at the 6 position of the 4H-1-benzopyran-4-one ring and with variation of the *para* substituent on the phenyl ring: (E)-6-chloro-3-[2-(4-chlorophenylsulfonyl)ethenyl]-4-chromanone, (I), (E)-6-bromo-3-[2-(4-bromophenylsulfonyl)ethenyl]-4-chromanone, (II), and (E)-3-[2-(4-chlorophenylsulfonyl)ethenyl]-6-methoxy-4-chromanone, (III). Our aim was to observe the influences of these changes upon the

conformation of the ethenylsulfone moiety. Comparisons may also be made with the three-dimensional structure of the antifungicide, (*E*)-3-[2-(4-chlorophenylsulfonyl)ethenyl]-4*H*-1-benzopyran-4-one, (IV), (*i.e.* *para*-chloro and 6-H) (Krishnaiah, Raju, Lu, Chen & Rao, 1995).



- (I) $R = \text{Cl}, R' = \text{Cl}$
 (II) $R = \text{Br}, R' = \text{Br}$
 (III) $R = \text{Cl}, R' = \text{OCH}_3$
 (IV) $R = \text{Cl}, R' = \text{H}$

The solid-state structures of molecules (I), (II) and (III) (Fig. 1) confirm the *trans* conformation at C(9)—C(10). Bond distances reflect electron delocalization in the O(1)—C(2)—C(3)—C(9)—C(10) chain. There are no significant differences in angles and distances for the three structures.

Molecules (I), (II) and (III) show coplanarity of the sulfur, ethene and chromanone ring moieties

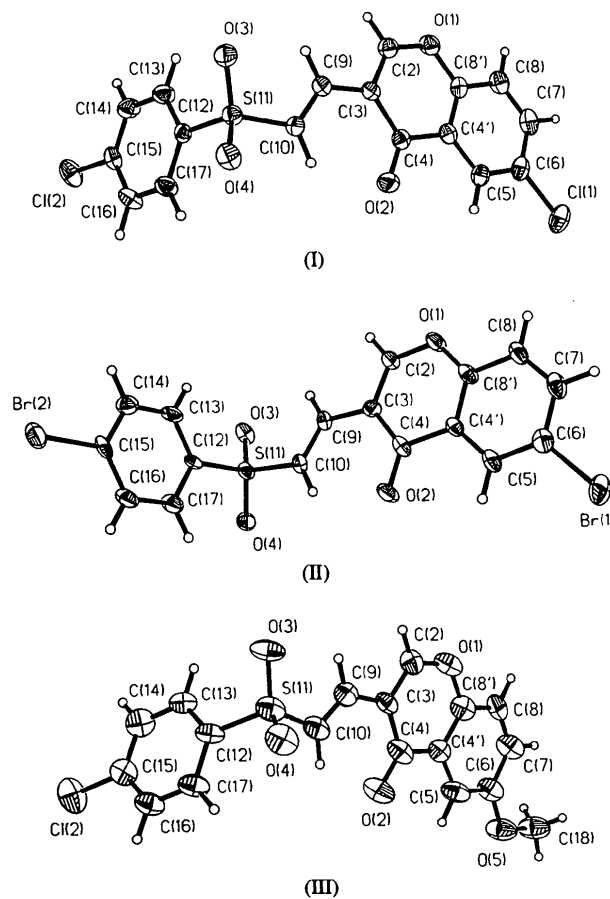


Fig. 1. Views of compounds (I), (II) and (III) with displacement ellipsoids shown at the 50% probability level.

[S(11),O(1),C(2)—C(10); deviations: 0.02 (I), 0.02 (II) and 0.01 Å (III)]. However, in the methoxy-substituted chromanone derivatives (III) and (IV), O(3) lies in that plane (deviation 0.05 Å), whereas in (I) and (II), O(3) is 0.569 and 0.548 Å, respectively, from that plane. Thus, the S(11),O(1),C(2)—C(10) plane, including O(3), bisects the C(12)—S(11)—O(4) angle in compounds (III) and (IV) as seen in a projection down the S(11)—C(10) bond, but is perpendicular to the S(11)—C(12) bond in the same projection for (I) and (II) [torsion angle C(9)—C(10)—S(11)—C(12): 99.0 (I), 98.8 (II), 123.1° (III)] (Fig. 2). This difference in conformation may be due to packing as (I) and (II) are isostructural and observed in a triclinic unit cell, whereas (III) and (IV) are found in a larger cell of monoclinic symmetry.

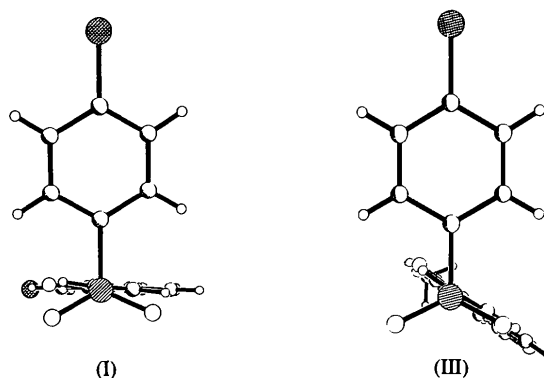


Fig. 2. Projection views of compounds (I) and (III) down the S(11)—C(10) bond.

Experimental

For the preparation of compounds (I), (II) and (III), a mixture of 6-*R'*-4-oxo-4*H*-1-benzopyran-3-carboxaldehyde (0.01 mol), 4-(*R*)phenylsulfonylacetic acid (0.01 mol) [(I) $R = \text{Cl}, R' = \text{Cl}$, (II) $R = \text{Br}, R' = \text{Br}$, (III) $R = \text{Cl}, R' = \text{OCH}_3$], 10 ml glacial acetic acid and a small amount of benzylamine (0.2 ml) was heated under reflux for 2 h. The reaction mixture was treated with 50 ml of anhydrous diethyl ether and allowed to stand for 18 h. The solid formed was crystallized from glacial acetic acid to yield the title compounds.

Compound (I)

Crystal data

$\text{C}_{17}\text{H}_{10}\text{Cl}_2\text{O}_4\text{S}$
 $M_r = 381.2$
 Triclinic
 $P\bar{1}$
 $a = 6.778 (1) \text{ \AA}$
 $b = 10.451 (1) \text{ \AA}$
 $c = 11.618 (1) \text{ \AA}$
 $\alpha = 91.34 (1)^\circ$
 $\beta = 98.88 (1)^\circ$
 $\gamma = 97.51 (1)^\circ$
 $V = 805.3 (2) \text{ \AA}^3$
 $Z = 2$
 $D_x = 1.572 \text{ Mg m}^{-3}$
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 65 reflections
 $\theta = 5.721\text{--}12.505^\circ$
 $\mu = 0.551 \text{ mm}^{-1}$
 $T = 298 \text{ K}$
 Needle
 $0.3 \times 0.2 \times 0.2 \text{ mm}$
 Colorless

Data collection

Syntex P4 four-circle diffractometer	$R_{\text{int}} = 0.018$
$\theta/2\theta$ scans	$\theta_{\text{max}} = 25.0^\circ$
Absorption correction: none	$h = -6 \rightarrow 6$
2006 measured reflections	$k = -10 \rightarrow 10$
1499 independent reflections	$l = 0 \rightarrow 11$
1292 observed reflections [$F > 4.0\sigma(F)$]	3 standard reflections
	frequency: 97 min
	intensity decay: 0.0001%

Refinement

Refinement on F	$w = 1/[\sigma^2(F) + 0.0008F^2]$
$R = 0.0308$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$wR = 0.0429$	$\Delta\rho_{\text{max}} = 0.22 \text{ e } \text{\AA}^{-3}$
$S = 1.14$	$\Delta\rho_{\text{min}} = -0.20 \text{ e } \text{\AA}^{-3}$
1499 reflections	Extinction correction: none
218 parameters	Atomic scattering factors
H atoms positioned by idealized geometry, riding model with fixed isotropic displacement parameters	from <i>International Tables for Crystallography</i> (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (I)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j.$$

	x	y	z	U_{eq}
S(11)	0.3009 (1)	0.5195 (1)	0.2219 (1)	0.040 (1)
Cl(1)	0.9485 (2)	-0.1030 (1)	-0.1887 (1)	0.071 (1)
Cl(2)	-0.1968 (2)	0.2150 (1)	0.5612 (1)	0.072 (1)
O(1)	0.2376 (3)	0.1646 (2)	-0.2373 (2)	0.048 (1)
O(2)	0.6347 (4)	0.2273 (2)	0.0671 (2)	0.058 (1)
O(3)	0.1664 (3)	0.5901 (2)	0.1490 (2)	0.050 (1)
O(4)	0.4792 (3)	0.5880 (2)	0.2907 (2)	0.055 (1)
C(2)	0.2124 (5)	0.2463 (3)	-0.1523 (3)	0.046 (1)
C(3)	0.3358 (5)	0.2734 (3)	-0.0488 (3)	0.039 (1)
C(4)	0.5158 (5)	0.2112 (3)	-0.0246 (3)	0.041 (1)
C(4')	0.5458 (5)	0.1226 (3)	-0.1192 (3)	0.040 (1)
C(5)	0.7147 (5)	0.0583 (3)	-0.1108 (3)	0.046 (1)
C(6)	0.7380 (5)	-0.0227 (3)	-0.1993 (3)	0.051 (1)
C(7)	0.5926 (7)	-0.0432 (3)	-0.3003 (3)	0.059 (2)
C(8)	0.4277 (6)	0.0195 (3)	-0.3106 (3)	0.054 (2)
C(8')	0.4053 (5)	0.1028 (3)	-0.2203 (3)	0.043 (1)
C(9)	0.2809 (5)	0.3670 (3)	0.0306 (3)	0.042 (1)
C(10)	0.3772 (5)	0.4024 (3)	0.1364 (3)	0.041 (1)
C(12)	0.1603 (5)	0.4325 (3)	0.3179 (3)	0.035 (1)
C(13)	-0.0444 (5)	0.4016 (3)	0.2873 (3)	0.050 (1)
C(14)	-0.1539 (5)	0.3339 (3)	0.3620 (3)	0.055 (1)
C(15)	-0.0575 (6)	0.2986 (3)	0.4660 (3)	0.046 (1)
C(16)	0.1475 (6)	0.3261 (4)	0.4970 (3)	0.055 (2)
C(17)	0.2573 (5)	0.3939 (3)	0.4220 (3)	0.049 (1)

Table 2. Selected geometric parameters (\AA , $^\circ$) for (I)

S(11)—O(3)	1.435 (2)	C(4')—C(5)	1.393 (5)
S(11)—O(4)	1.436 (2)	C(4')—C(8')	1.385 (4)
S(11)—C(10)	1.737 (4)	C(5)—C(6)	1.358 (5)
S(11)—C(12)	1.769 (3)	C(6)—C(7)	1.404 (5)
Cl(1)—C(6)	1.737 (4)	C(7)—C(8)	1.359 (6)
Cl(2)—C(15)	1.741 (4)	C(8)—C(8')	1.387 (5)
O(1)—C(2)	1.336 (4)	C(9)—C(10)	1.320 (4)
O(1)—C(8')	1.370 (4)	C(12)—C(13)	1.371 (5)
C(2)—C(3)	1.355 (4)	C(12)—C(17)	1.379 (4)
C(3)—C(4)	1.450 (5)	C(13)—C(14)	1.377 (5)
C(3)—C(9)	1.451 (5)	C(14)—C(15)	1.365 (5)
C(4)—C(4')	1.477 (5)	C(15)—C(16)	1.371 (5)
C(4)—O(2)	1.226 (4)	C(16)—C(17)	1.381 (5)

Compound (II)

Crystal data

C ₁₇ H ₁₀ Br ₂ O ₄ S
$M_r = 470.1$
Triclinic
$P\bar{1}$
$a = 6.761 (1) \text{\AA}$
$b = 10.719 (1) \text{\AA}$
$c = 11.744 (1) \text{\AA}$
$\alpha = 91.20 (1)^\circ$
$\beta = 99.10 (1)^\circ$
$\gamma = 97.71 (1)^\circ$
$V = 832.1 (2) \text{\AA}^3$
$Z = 2$
$D_x = 1.876 \text{ Mg m}^{-3}$
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073 \text{\AA}$
Cell parameters from 60 reflections
$\theta = 5.952\text{--}12.424^\circ$
$\mu = 5.015 \text{ mm}^{-1}$
$T = 298 \text{ K}$
Cube
$0.2 \times 0.2 \times 0.2 \text{ mm}$
Colorless

Data collection

Syntex P4 four-circle diffractometer	$R_{\text{int}} = 0.016$
$\theta/2\theta$ scans	$\theta_{\text{max}} = 25.0^\circ$
Absorption correction: none	$h = -6 \rightarrow 6$
2090 measured reflections	$k = -10 \rightarrow 10$
1560 independent reflections	$l = 0 \rightarrow 11$
1229 observed reflections [$F > 4.0\sigma(F)$]	3 standard reflections
	frequency: 97 min
	intensity decay: 0.0001%

Refinement

Refinement on F	$w = 1/[\sigma^2(F) + 0.0008F^2]$
$R = 0.0430$	$(\Delta/\sigma)_{\text{max}} = 0.002$
$wR = 0.0484$	$\Delta\rho_{\text{max}} = 0.49 \text{ e } \text{\AA}^{-3}$
$S = 1.18$	$\Delta\rho_{\text{min}} = -0.62 \text{ e } \text{\AA}^{-3}$
1560 reflections	Extinction correction: none
217 parameters	Atomic scattering factors
H atoms positioned by idealized geometry, riding model with fixed isotropic displacement parameters	from <i>International Tables for Crystallography</i> (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (II)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j.$$

	x	y	z	U_{eq}
Br(1)	-0.9473 (2)	0.6046 (1)	0.1881 (1)	0.064 (1)
Br(2)	0.2072 (2)	0.2853 (1)	0.4384 (1)	0.063 (1)
S(11)	-0.3065 (4)	-0.0191 (2)	0.7804 (2)	0.037 (1)
O(1)	-0.2295 (11)	0.3253 (7)	1.2352 (6)	0.046 (3)
O(2)	-0.6316 (12)	0.2705 (7)	0.9346 (7)	0.056 (3)
O(3)	-0.1740 (11)	-0.0894 (6)	0.8523 (6)	0.047 (3)
O(4)	-0.4880 (10)	-0.0839 (7)	0.7120 (6)	0.049 (3)
C(2)	-0.2043 (17)	0.2463 (10)	1.1523 (9)	0.041 (4)
C(3)	-0.3319 (15)	0.2208 (10)	1.0499 (9)	0.036 (4)
C(4)	-0.5081 (18)	0.2844 (10)	1.0252 (9)	0.041 (5)
C(4')	-0.5365 (16)	0.3709 (9)	1.1203 (9)	0.038 (4)
C(5)	-0.7013 (18)	0.4373 (10)	1.1107 (9)	0.046 (5)
C(6)	-0.7219 (18)	0.5170 (10)	1.1989 (11)	0.048 (5)
C(7)	-0.5776 (20)	0.5346 (11)	1.2985 (10)	0.054 (5)
C(8)	-0.4141 (19)	0.4706 (10)	1.3087 (9)	0.048 (5)
C(8')	-0.3917 (18)	0.3877 (10)	1.2196 (9)	0.041 (4)
C(9)	-0.2829 (16)	0.1276 (9)	0.9696 (9)	0.037 (4)
C(10)	-0.3825 (16)	0.0950 (9)	0.8651 (9)	0.036 (4)
C(12)	-0.1655 (15)	0.0652 (8)	0.6863 (8)	0.030 (4)
C(13)	0.0407 (17)	0.0950 (10)	0.7162 (9)	0.048 (5)
C(14)	0.1528 (18)	0.1620 (11)	0.6434 (10)	0.053 (5)

C(15)	0.0538 (19)	0.1962 (10)	0.5389 (9)	0.043 (5)	O(2)	0.0473 (2)	0.3347 (8)	0.4357 (3)	0.093 (2)
C(16)	-0.1540 (19)	0.1657 (11)	0.5086 (10)	0.053 (5)	O(3)	0.16347 (15)	-0.1597 (6)	0.5627 (3)	0.093 (2)
C(17)	-0.2616 (18)	0.1023 (10)	0.5831 (10)	0.051 (5)	O(4)	0.16636 (14)	0.1322 (7)	0.6790 (3)	0.085 (2)

Table 4. Selected geometric parameters (\AA , $^\circ$) for (II)

Br(2)—C(15)	1.884 (12)	C(5)—C(6)	1.365 (17)	O(2)	0.0473 (2)	0.3347 (8)	0.4357 (3)	0.093 (2)
Br(1)—C(6)	1.884 (12)	C(6)—C(7)	1.392 (16)	O(3)	0.16347 (15)	-0.1597 (6)	0.5627 (3)	0.093 (2)
S(11)—O(3)	1.433 (8)	C(7)—C(8)	1.368 (19)	O(4)	0.16636 (14)	0.1322 (7)	0.6790 (3)	0.085 (2)
S(11)—O(4)	1.439 (7)	C(8)—C(8')	1.398 (16)	O(5)	-0.0844 (2)	0.4738 (8)	0.2929 (4)	0.106 (2)
S(11)—C(10)	1.741 (11)	C(2)—C(3)	1.363 (14)	C(2)	0.0336 (3)	-0.1901 (12)	0.4054 (5)	0.069 (2)
S(11)—C(12)	1.758 (10)	C(3)—C(9)	1.465 (15)	C(3)	0.0519 (2)	-0.0168 (10)	0.4321 (5)	0.059 (2)
O(2)—C(4)	1.238 (13)	C(9)—C(10)	1.319 (13)	C(4)	0.0325 (2)	0.1718 (12)	0.4158 (4)	0.072 (2)
O(1)—C(8')	1.350 (14)	C(12)—C(13)	1.375 (15)	C(4')	-0.0058 (2)	0.1553 (11)	0.3721 (4)	0.060 (2)
O(1)—C(2)	1.323 (13)	C(12)—C(17)	1.374 (14)	C(5)	-0.0286 (3)	0.3209 (11)	0.3507 (5)	0.083 (3)
C(4)—C(4')	1.488 (15)	C(13)—C(14)	1.381 (17)	C(6)	-0.0652 (3)	0.3014 (13)	0.3104 (5)	0.079 (3)
C(4)—C(3)	1.444 (16)	C(14)—C(15)	1.383 (15)	C(7)	-0.0800 (3)	0.1100 (13)	0.2891 (5)	0.085 (3)
C(4')—C(5)	1.393 (17)	C(15)—C(16)	1.386 (17)	C(8)	-0.0588 (3)	-0.0519 (13)	0.3105 (5)	0.068 (2)
C(4')—C(8')	1.390 (14)	C(16)—C(17)	1.361 (17)	C(8')	-0.0225 (3)	-0.0319 (12)	0.3508 (5)	0.072 (2)
				C(9)	0.0902 (2)	-0.0319 (10)	0.4751 (4)	0.065 (2)
				C(10)	0.1143 (2)	0.1071 (10)	0.5150 (4)	0.065 (2)
				C(12)	0.1819 (2)	0.1868 (9)	0.5061 (4)	0.062 (2)
				C(13)	0.1888 (2)	0.1053 (10)	0.4194 (4)	0.065 (2)
				C(14)	0.2065 (2)	0.2173 (12)	0.3639 (4)	0.080 (2)
				C(15)	0.2169 (2)	0.4099 (11)	0.3941 (5)	0.071 (2)
				C(16)	0.2097 (2)	0.4904 (10)	0.4793 (5)	0.073 (2)
				C(17)	0.1917 (2)	0.3814 (9)	0.5348 (4)	0.071 (2)
				C(18)	-0.1222 (3)	0.4625 (12)	0.2469 (6)	0.110 (3)

Compound (III)*Crystal data*C₁₈H₁₃ClO₅SM_r = 376.79

Monoclinic

C2/c

a = 39.053 (13) \AA b = 6.700 (2) \AA c = 13.471 (5) \AA β = 106.62 (3) $^\circ$ V = 3378 (2) \AA^3

Z = 8

D_x = 1.482 Mg m⁻³D_m not measured*Data collection*

Syntex P4 four-circle diffractometer

 $\theta/2\theta$ scans

Absorption correction: none

2685 measured reflections

2166 independent reflections

2129 observed reflections

 $[F > 2\sigma(F)]$ *Refinement*Refinement on F^2

R(F) = 0.0687

wR(F²) = 0.1612

S = 1.14

2129 reflections

227 parameters

H atoms positioned by idealized geometry, riding model with fixed isotropic displacement parameters

Mo K α radiation λ = 0.71073 \AA

Cell parameters from 68 reflections

 θ = 6.335–12.465 $^\circ$ μ = 0.376 mm⁻¹

T = 298 K

Cube

0.2 \times 0.2 \times 0.2 mm

Colorless

R_{int} = 0.088 θ_{max} = 24.99 $^\circ$ h = -25 \rightarrow 25k = 0 \rightarrow 7l = 0 \rightarrow 16

3 standard reflections

frequency: 97 min

intensity decay: 0.0001%

 $w = 1/[\sigma^2(F_o^2) + (0.0985P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = -0.001$ $\Delta\rho_{\text{max}} = 0.335 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.229 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

Atomic scattering factors

from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)Table 5. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (III)
$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	U _{eq}
Cl(2)	0.23830 (7)	0.5493 (4)	0.32364 (14)	0.1073 (10)
S(11)	0.15766 (6)	0.0501 (3)	0.57523 (12)	0.0705 (7)
O(1)	-0.0019 (2)	-0.2031 (7)	0.3675 (3)	0.081 (2)

Table 6. Selected geometric parameters (\AA , $^\circ$) for (III)

Cl(2)—C(15)	1.711 (8)	C(8')—C(8)	1.369 (10)
S(11)—O(3)	1.442 (5)	C(8')—C(4')	1.406 (10)
S(11)—O(4)	1.450 (4)	C(8)—C(7)	1.345 (10)
S(11)—C(10)	1.698 (8)	C(7)—C(6)	1.401 (10)
S(11)—C(12)	1.763 (7)	C(6)—C(5)	1.384 (11)
O(2)—C(4)	1.228 (7)	C(5)—C(4')	1.401 (10)
O(1)—C(2)	1.336 (9)	C(9)—C(10)	1.324 (9)
O(1)—C(8')	1.382 (9)	C(12)—C(17)	1.383 (8)
O(5)—C(6)	1.360 (9)	C(12)—C(13)	1.385 (8)
O(5)—C(18)	1.431 (11)	C(17)—C(16)	1.372 (9)
C(4)—C(4')	1.446 (10)	C(16)—C(15)	1.368 (9)
C(4)—C(3)	1.457 (10)	C(15)—C(14)	1.379 (9)
C(3)—C(2)	1.357 (9)	C(14)—C(13)	1.376 (9)
C(3)—C(9)	1.446 (10)		

Variable scan rate, θ - 2θ scan mode with a scan width of 0.6 $^\circ$ below K α_1 and 0.6 $^\circ$ above K α_2 to a maximum 2θ value of 50 $^\circ$. Refinement was completed using full-matrix least-squares methods.

For all compounds, data collection: XSCANS (Siemens, 1991); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structures: SHELXS86 (Sheldrick, 1990); program(s) used to refine structures: SHELXS86; molecular graphics: XP (Siemens, 1990)

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1224). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CHI 2HU, England.

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