

The Crystal Structure of an Anthracene Sulfur Mustard, 9-[[2-Chloroethyl]thio]methyl]anthracene

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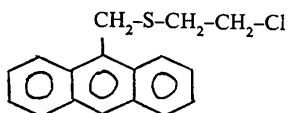
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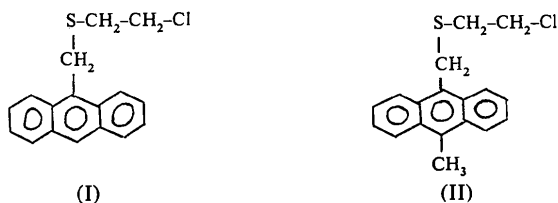
The aromatic alkylating agent, 9-[[2-chloroethyl]thio]methyl]anthracene, $C_{17}H_{15}ClS$, with antitumor properties, crystallizes in the orthorhombic space group $Pcab$ with $Z=8$, $a=16.569$ (10), $b=22.923$ (18), $c=7.688$ (5) Å, $V=2920.0$ (9) Å³, $D_x=1.26$ and $D_m=1.25$ g cm⁻³. The structural formula is:



Three-dimensional diffractometer data, collected with monochromatic Cu $K\alpha$ radiation, consisted of 2770 reflections, of which 2146 were above the threshold of $2\sigma(I)$. The structure was solved by the vector superposition method and refined by a full-matrix least-squares procedure to a final $R=0.058$. All hydrogen atoms were located and refined isotropically. There is an angle of 4.8° between the planes of the two outer benzene rings, each of which is planar within experimental error. The packing around the $-S-CH_2-CH_2-Cl$ group is very similar to that for the analogue with a methyl group in the 10-position. This packing is probably a significant determinant of the crystal structure since the stacking of aromatic ring systems in planes 3.4 Å apart, found for the methyl analogue, does not occur in the structure reported here.

Introduction

Many alkylating agents, such as nitrogen mustard [$CH_3N(CH_2CH_2Cl)_2$], show antitumor activity. The compound 9-[[2-chloroethyl]thio]methyl]anthracene (I) was prepared by Dr Richard Peck of this Institute.



It was found to be a mild antitumor agent against the ascites form of Ehrlich carcinoma (Peck, O'Connell & Creech, 1967, 1970). It is a monofunctional sulfur mustard derivative of anthracene, and was prepared and tested because of its analogy to the monofunctional nitrogen mustards that are active against

tumors (Creech, Preston, Peck, O'Connell & Ames, 1972).

It has been suggested that the monofunctional half-mustards, with only one alkylating $-CH_2-CH_2Cl$ group, have a bifunctional mechanism (Peck & O'Connell, 1972). The 2-chloroethyl group is believed to react chemically with a base of DNA (such as a guanine group) (Lawley & Wallick, 1957), while the polycyclic component interacts with DNA by intercalation between the base pairs of DNA as proposed by Lerman (1964) for the acridines and Boyland & Green (1962) for hydrocarbons. It has been shown (Peck & O'Connell, 1972; Peck, Tan & Peck, 1976) that there is little correlation between alkylating capabilities and antitumor capabilities. This result suggests that the bifunctional mechanism is initiated by the intercalation of the polycyclic portion of the molecule in DNA and that alkylation is a terminal part of this mechanism (Ames & Whitfield, 1966; Peck *et al.*, 1967).

The compound (I) was studied in order to compare its molecular coordinates with those of the 10-methyl analogue of known crystal structure (II) (Glusker & Zacharias, 1972) which had enhanced antitumor activity (Peck *et al.*, 1967). It was felt that the additional methyl group might be responsible for the observed stacking in the crystalline state.

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Experimental

Light yellow crystalline needles of (I) were grown from ethanol from material kindly provided by Dr R. M. Peck. One needle was cut to a size of $0.18 \times 0.21 \times 0.24$ mm for the data collection. The cell data are given in Table 1. Intensity data were collected on an automated four-circle diffractometer with monochromatized Cu $K\alpha$ radiation using the θ - 2θ variable scan technique. A total of 2770 independent reflections were scanned of which 2146 were above the assigned threshold value of $2\sigma(I)$ [where $\sigma(I)$ was found from counting statistics]. A remeasurement of the most intense reflections after the completion of the data collection showed that some intensity changes had occurred during the data collection. Of the 113 most intense reflections, 20 changed significantly (9 increased, 11 decreased in intensity). There was some evidence that the lattice parameters increased during the data collection, probably due to X-ray damage. The increase in V was $4\sigma(V)$. However, the intensity fall-off during data collection as indicated by the standard reflections, was negligible. Values of $\sigma(F)$ for observed data were calculated from the formula $\sigma(F) = (F/2) \{[\sigma^2(I)/(I)^2] + \delta^2\}^{1/2}$ where δ is a measured

instrumental uncertainty, 0.0150, determined from the variation of the intensities of periodically monitored check reflections. An absorption correction (Johnson, 1963) was applied. The density of the crystal was measured in a potassium iodide solution.

Structure solution

The structure was solved by the vector superposition method starting with a high peak, the chlorine-sulfur vector, found in the Patterson map. A sixfold superposition on symmetry-related vectors produced a trial structure with an initial residual of 0.33. After three cycles of isotropic full-matrix least-squares refinement the R value was reduced to 0.24. Three cycles of full-matrix least-squares anisotropic refinement gave $R = 0.091$. The positions of the hydrogen atoms were derived from difference Fourier syntheses and refinement of all the atoms was continued with hydrogen atoms treated isotropically. The quantity minimized was $\sum \omega \{ |F_o| - |F_c| \}^2$ and the weights, ω , for the final refinement were $[1/\sigma^2(F)]$; reflections with $I < 2\sigma(I)$ were assigned weights of zero. The final refinement resulted in a residual, R , of 0.058 and a weighted R value of 0.064.*

The atomic scattering factors used for chlorine, sulfur and carbon were those listed in *International Tables for X-ray Crystallography* (1962). For hydrogen atoms the values given by Stewart, Davidson &

Table 1. *Crystal data for*
9- $\{[(2\text{-chloroethylthio})\text{methyl}]\}$ anthracene

Formula: C ₁₇ H ₁₅ ClS	Formula weight: 286.5
Crystal system: orthorhombic	Space group: <i>Pcab</i>
$a = 16.569$ (10) Å	$Z = 8$
$b = 22.923$ (18)	$F(000) = 1200$
$c = 7.688$ (5)	$\mu(\text{Cu } K\alpha) = 32.2 \text{ cm}^{-1}$
$V = 2920.0$ (35) Å ³	$\lambda(\text{Cu } K\alpha) = 1.5418 \text{ Å}$
$D_x = 1.26 \text{ g cm}^{-3}$	
$D_m = 1.25$ (in aqueous KI)	

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31612 (12 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. *Final atomic parameters*

Positional parameters are given as fractional cell coordinates. Anisotropic temperature factors are expressed as: $\exp[-\frac{1}{2}(h^2 a^{*2} B_{11} + k^2 b^{*2} B_{22} + l^2 c^{*2} B_{33} + 2hka^*b^*B_{12} + 2hla^*c^*B_{13} + 2klb^*c^*B_{23})]$; and isotropic temperature factors as: $\exp(-B \sin^2 \theta/\lambda^2)$ with B_{ij} and B values given in Å². The standard deviations for each parameter, determined from the inverted full matrix, are given in parentheses and apply to the last specified digits.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cl	0.14397 (10)	0.01508 (5)	1.13250 (13)	15.08 (8)	9.53 (5)	4.76 (4)	7.16 (7)	0.25 (5)	2.05 (4)
S	0.08263 (6)	0.14299 (4)	0.73626 (9)	8.54 (5)	4.88 (3)	3.20 (3)	2.06 (4)	-0.74 (3)	-0.05 (3)
C(1)	0.0893 (2)	0.0528 (2)	0.9625 (5)	7.19 (20)	6.67 (17)	4.65 (15)	1.83 (17)	0.51 (15)	0.94 (14)
C(2)	0.1404 (2)	0.0997 (2)	0.8896 (4)	5.56 (16)	6.97 (17)	3.65 (13)	1.17 (15)	-0.35 (13)	-0.19 (13)
C(3)	0.0942 (2)	0.1004 (1)	0.5389 (4)	5.42 (15)	3.57 (10)	3.12 (11)	1.16 (11)	-0.30 (11)	0.27 (9)
C(4)	0.0486 (2)	0.1305 (1)	0.3944 (3)	4.19 (12)	3.13 (9)	2.76 (10)	0.87 (9)	-0.01 (9)	0.03 (8)
C(5)	0.0870 (2)	0.1762 (1)	0.3018 (3)	3.57 (11)	3.40 (10)	2.77 (9)	0.63 (9)	0.06 (9)	-0.18 (8)
C(6)	0.1674 (2)	0.1960 (1)	0.3378 (4)	3.99 (13)	5.44 (13)	4.04 (13)	0.68 (11)	-0.37 (11)	0.32 (12)
C(7)	0.2012 (2)	0.2401 (2)	0.2462 (4)	4.08 (14)	6.44 (16)	5.17 (15)	-1.06 (13)	0.21 (13)	0.22 (15)
C(8)	0.1583 (2)	0.2687 (2)	0.1125 (5)	5.36 (16)	5.25 (14)	5.12 (16)	-0.86 (13)	0.58 (14)	1.08 (14)
C(9)	0.0832 (2)	0.2515 (1)	0.0724 (4)	5.35 (15)	4.25 (12)	3.79 (11)	0.27 (12)	0.14 (12)	0.94 (11)
C(10)	0.0446 (2)	0.2049 (1)	0.1636 (3)	3.80 (12)	3.35 (10)	2.99 (10)	0.59 (9)	0.37 (10)	0.18 (9)
C(11)	-0.0336 (2)	0.1877 (1)	0.1224 (3)	4.22 (13)	3.84 (11)	3.06 (10)	0.85 (10)	-0.34 (10)	0.22 (9)
C(12)	-0.0723 (2)	0.1441 (1)	0.2144 (3)	3.91 (12)	3.47 (10)	3.36 (10)	0.48 (9)	-0.02 (9)	-0.53 (9)
C(13)	-0.1538 (2)	0.1281 (2)	0.1756 (5)	4.41 (14)	5.16 (14)	4.84 (15)	0.17 (12)	-0.68 (12)	-0.57 (13)
C(14)	-0.1927 (2)	0.0876 (2)	0.2734 (5)	4.47 (14)	5.71 (15)	6.69 (20)	-1.32 (13)	0.08 (15)	-0.93 (16)
C(15)	-0.1531 (2)	0.0600 (1)	0.4127 (5)	6.06 (18)	4.38 (13)	5.81 (17)	-1.01 (13)	1.14 (15)	-0.23 (14)
C(16)	-0.0758 (2)	0.0730 (1)	0.4524 (4)	5.86 (17)	3.41 (11)	4.15 (13)	-0.14 (12)	0.53 (13)	-0.01 (10)
C(17)	-0.0311 (2)	0.1155 (1)	0.3554 (3)	4.42 (13)	2.88 (9)	3.03 (10)	0.40 (9)	0.30 (10)	-0.35 (9)

Table 2 (*cont.*)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
H(1)	0.0400 (22)	0.0712 (15)	1.0158 (51)	6.0 (9)
H(1')	0.0750 (26)	0.0202 (20)	0.8690 (62)	8.5 (12)
H(2)	0.1921 (22)	0.0763 (16)	0.8331 (53)	7.2 (9)
H(2')	0.1547 (24)	0.1243 (19)	0.9905 (60)	7.8 (11)
H(3)	0.1545 (19)	0.0965 (13)	0.5141 (41)	4.1 (7)
H(3')	0.0735 (18)	0.0648 (14)	0.5547 (44)	5.0 (8)
H(6)	0.1968 (17)	0.1781 (13)	0.4268 (41)	4.1 (6)
H(7)	0.2572 (23)	0.2512 (15)	0.2733 (44)	5.9 (8)
H(8)	0.1822 (21)	0.3000 (17)	0.0475 (56)	6.8 (10)
H(9)	0.0556 (19)	0.2678 (14)	-0.0154 (45)	4.9 (8)
H(11)	-0.0611 (19)	0.2086 (13)	0.0263 (47)	5.3 (8)
H(13)	-0.1816 (19)	0.1487 (14)	0.0785 (47)	5.1 (8)
H(14)	-0.2432 (21)	0.0793 (14)	0.2455 (45)	5.1 (8)
H(15)	-0.1806 (22)	0.0323 (17)	0.4745 (53)	6.4 (10)
H(16)	-0.0527 (19)	0.0575 (14)	0.5632 (49)	5.2 (8)

Simpson (1965) were used. The real components of anomalous dispersion correction for chlorine, and sulfur, 0.348 and 0.319 respectively, are those listed by Cromer & Liberman (1970). Computer programs used were *UCLALS4* (Gantzel, Sparks, Long & Trueblood, 1969), modified by H. L. Carrell, and the *CRYSNET* package (Bernstein *et al.*, 1974). The final atomic parameters are presented in Table 2.

Discussion of structure

The interatomic distances and interbond angles in this molecule are shown in Fig. 1. The following is a comparison of the molecular geometries of (I) and its 10-methyl analogue (II):

1. The angle C(10)–C(11)–C(12) is increased from 119.2 (3)° in (II) to 121.5 (3)° in (I) when a methyl group is replaced by a hydrogen atom on C(11) [see Fig. 1 and Table 3(a)]. The relationship of such an angle in a benzene ring to the electronegativity of the substituent has been pointed out by Domenicano, Mazzeo & Vaciego (1975).

2. The distance C(1)–C(2) is decreased and C(1)–Cl increased suggesting that (I) (without a methyl group) might be a better alkylating agent (since the form $-\text{CH}_2^+\text{Cl}^-$, with the C–Cl bond already broken, might contribute more to the structure [see Fig. 1 and Table 3(a)]).

3. Angles between the planar outer rings of the anthracene ring system are 4.8° for (I) (no methyl group) and 8.7° for (II) (methyl group) [see Table 3(b), (c) and (d)]. This non-planarity of the anthracene ring system results mainly from effects in the central ring. The smaller angle at C(11) (representing a decrease of 2.3° on methylation) in (II) must have caused additional puckering increasing the nonplanarity of the system.

4. The general shapes of the two molecules [excluding the additional methyl group in (II)] are very similar, as shown in Fig. 2.

5. The packing of the ring systems is quite different

Table 3. *Some molecular parameters for 9-[[2-chloroethyl)thio]methyl}anthracene (I) and 10-methyl-9-[[2-chloroethyl)thio]methyl}anthracene (II)*

	(I) (this work)	(II) (additional methyl group)
(a) Bond distances and angles		
C(1)–Cl	1.809 (4) Å	1.792 (3) Å
C(1)–C(2)	1.478 (5)	1.501 (4)
C(10)–C(11)–C(12)	121.5 (3)°	119.2 (3)°
(b) Angles between planes		
A–C	4.83°	8.66°
A–B	3.26	5.24
B–C	1.57	3.42
(c) R.m.s. deviation from planes		
ABC	0.046 Å	0.317 Å
A	0.006	0.010
B	0.012	0.029
C	0.005	0.007
(d) Sum of internal angles in rings		
A	719.9°	719.9°
B	719.9	719.5
C	720.1	720.0

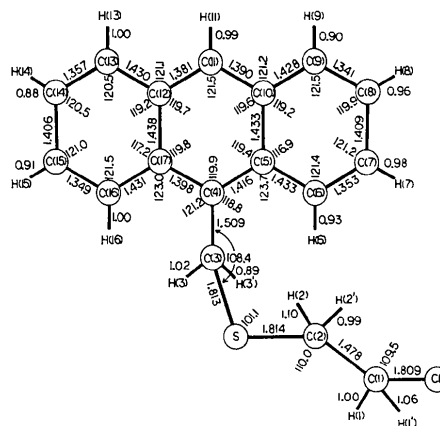


Fig. 1. Distances (Å) and angles (°). Estimated standard deviations are: 0.003–0.004 Å for C–Cl and C–S bonds; 0.004–0.005 Å for C–C bonds; 0.03–0.05 Å for C–H bonds; 0.2–0.3° for angles, except for those containing hydrogen atoms when the e.s.d. is 2–3°.

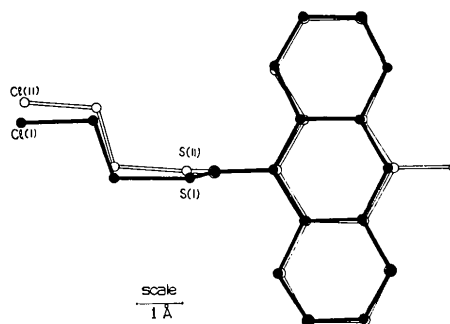


Fig. 2. Comparison of shapes of molecules (I) and (II). Black circles (I) (this work), open circles (II).

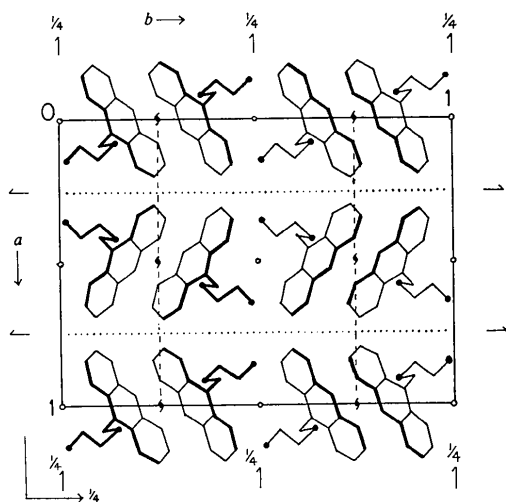


Fig. 3. Packing in unit cell. The side of the molecule with higher z values is indicated by heavier lines. Note that the anthracene ring systems do not stack.

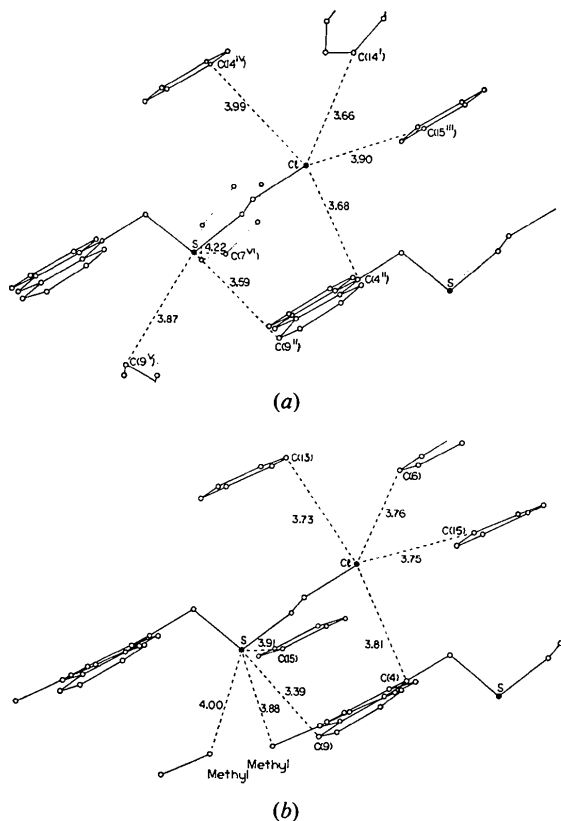


Fig. 4. Packing around sulfur and chlorine atoms in (I) and (II). (a) Packing in (I). (b) Packing in (II). Note the great similarity of the packing around sulfur and chlorine atoms in the two structures. These diagrams are viewed onto the plane of C(2)–S–C(3) in each case. [Note: C(13)–C(18) in (II) have been renumbered C(12)–C(17) to conform with the numbering of (I).] The symmetry codes for molecules (i) through (vi) are: (i) $\frac{1}{2} + x, -y, 1\frac{1}{2} - z$; (ii) $x, y, 1 + z$; (iii) $-x, -y, 2 - z$; (iv) $-x, -y, 1 - z$; (v) $-x, \frac{1}{2} - y, \frac{1}{2} + z$; (vi) $\frac{1}{2} - x, y, \frac{1}{2} + z$.

for the two molecules. In crystals of molecule (II) the anthracene rings overlap in planes 3.4 Å apart across a center of symmetry, with the 10-methyl group taking part in this packing. In crystals of molecule (I) there is no such stacking of ring systems, as shown in Fig. 3.

The packing around sulfur and chlorine atoms shows many similarities in the two structures as shown in Fig. 4(a) (I) and (b) (II) and may be a significant determinant of the crystal structure. In (I) [Fig. 4(a)] the closest group to the sulfur atom is C(9) of other molecules, one at a distance of 3.59 Å [with C(10) and C(11) also close as shown in Table 4(b)] and the other at a distance of 3.87 Å. In (II) [Fig. 4(b)] the closest group is also C(9) of another molecule at a shorter distance of 3.39 Å, other packing occurring at 3.88–4.00 Å with two methyl groups and C(14). It has been pointed out by Rosenfield & Parthasarathy (1975) that there are directional preferences for non-bonded atomic contacts with sulfur. They showed that groups (e.g. Cl⁻) that can donate electrons to sulfur tend to be in the C–S–C plane while groups (e.g. H⁺) that can accept electrons from sulfur tend to lie perpendicular to this direction. Using these criteria the experimental results for this compound (see Fig. 4) indicate that C(9'') tends to donate electrons to the sulfur atom, indicating a residual negative charge on C(9'').

In general the packing around the chlorine atoms in both structures is remarkably similar. Each chlorine atom, as shown in Fig. 4, is surrounded by four molecules at 3.66–3.99 Å for (I) and 3.73–3.81 Å for (II). In both structures C(9) of one molecule packs close to the sulfur atom of another molecule and C(4) of the first molecule also packs close to the chlorine atom of the second molecule. Other closest contacts with other molecules are *via* C(14), C(14) and C(15) for (I) and *via* C(6), C(13) and C(15) in (II). Thus the contacts around the chlorine atom common to both structures (I) and (II) are *via* C(4) [3.68 Å in (I) and 3.81 Å in (II)] and C(15) [3.90 Å in (I) and 3.75 Å in (II)].

Thus in (I) (without a methyl group), compared with (II), there is a slightly looser packing around the sulfur atom, similar packing around the chlorine atom, and an elimination of the tendency of anthracene rings to stack in the crystalline state.

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The Crystal Structures of RbZnSO₄Cl and TlZnSO₄Cl

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From single-crystal X-ray diffractometer data the structure of RbZnSO₄Cl has been determined. A refinement of the structure of TlZnSO₄Cl, based on diffractometer data, is also given. The compounds are closely related, crystallizing in the monoclinic space group $P2_1/c$ with $Z=4$. The structure of the Rb compound may, however, be disordered. The cell dimensions are $a=7.2610$ (3), $b=9.6152$ (5), $c=8.3086$ (6) Å, $\beta=95.542$ (6)° and $a=7.278$ (1), $b=9.551$ (1), $c=8.092$ (1) Å, $\beta=93.97$ (1)° for the Rb and Tl compounds respectively. Fundamental building elements of the structures are infinite layers of formula $(ZnSO_4Cl)_n^{2-}$, parallel to the bc plane. The layers are held together by Rb⁺ or Tl⁺ ions. There seems to be no reason to assume the presence of a stereochemical inert pair for Tl⁺.

Introduction

The present investigation was undertaken to study the coordination polyhedra of univalent Tl and Rb. A preliminary study of TlZnSO₄Cl has been published (Bosson, 1973). Work is in progress on the structures of Tl₃[Hg(SO₄)₂] [HgSO₄Cl], Rb₃[Hg(SO₄)₂] [HgSO₄Cl] and Tl₂S₂O₃.

Experimental

Single crystals of RbZnSO₄Cl and TlZnSO₄Cl were prepared by melting MCl (M=Rb or Tl) and anhydrous ZnSO₄ in the mole ratio 1:1 at 440°C in porcelain crucibles (Luzhnaya & Vereshchetne, 1956). The preparations consisted of colourless, transparent thin plates. The homogeneities of the samples were checked by Guinier-Hägg powder photographs.

RbZnSO₄Cl

Weissenberg photographs indicated the space group $P2_1/c$ as the following spectra were systematically absent: $h0l$, $l \neq 2n$; $0k0$, $k \neq 2n$. However, horizontal streaks occurred in the photographs. A few very weak $h0l$ reflexions for $l=2n+1$ were recorded on the diffractometer. They are most probably due to parasites but a disordering cannot be excluded.

The lattice constants were obtained from a least-squares analysis of the settings of 40 reflexions measured on the diffractometer with Cu $K\alpha_1$ radiation ($\lambda=1.54051$ Å). The density was determined by displacement in benzene. Some crystal data are given in Table 1.

A single crystal, 0.04 × 0.22 × 0.23 mm along **a**, **b** and **c**, respectively, was selected and mounted so that a crystallographic axis did not coincide with the goniometer ϕ -axis.