

Action of Heat on 6-*p*-Chlorophenylsulphonylamino-5-*p*-chlorophenylsulphonylimino-1,2,5,6-tetrahydro-4,6-butano-4*H*-pyrrolo[3,2,1-*ij*]quinoline: Crystal Structure of 7*a-p*-Chlorophenylsulphonylamino-4,5,7*a*,8,9,10-hexahydro-7*H*-pyrrolo[3,2,1-*de*]phenanthridine

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On heating to 130 °C, 6-*p*-chlorophenylsulphonylamino-5-*p*-chlorophenylsulphonylimino-1,2,5,6-tetrahydro-4,6-butano-4*H*-pyrrolo[3,2,1-*ij*]quinoline rearranges forming 7,7*a*-bis-(*p*-chlorophenylsulphonylamino)-4,5,7*a*,8,9,10-hexahydro-7*H*-pyrrolo[3,2,1-*de*]phenanthridine. Reduction of this compound with borohydride results in elimination of one of the *p*-chlorophenylsulphonylamino groups. The crystal structure of 7*a-p*-chlorophenylsulphonylamino-4,5,7*a*,8,9,10-hexahydro-7*H*-pyrrolo[3,2,1-*de*]phenanthridine ($a = 11.93$, $b = 10.088$, $c = 16.23$ Å, $\beta = 104.27^\circ$; space group $P2_1/c$; four-circle diffractometer data; 2 789 independent reflections; Mo- K_α radiation) has been determined by direct methods and refined by block matrix least-squares to $R = 0.036$ in order to establish the molecular structure.

THE bridged compound [I; Z = tosyl (Ts) or *p*-chlorophenylsulphonyl (Cbs)] is smoothly transformed¹ into the indole (III) *via* compound (II) when it is dissolved in trifluoroacetic acid (TFA). The same transformation occurs in high yield on melting compound (I). In contrast compound (IV) forms an ion (V) when dissolved in TFA and this ion reacts with ethanol to form compound (VI).² The trifluoroacetate salt of the ion (V; Z = Ts) has now been obtained crystalline; it contains two molecules of TFA, one of which may have added across the C=N⁺ bond; similar reactions have been observed³

¹ A. S. Bailey and P. A. Wilkinson, *J.C.S. Perkin I*, 1976, 481.

² A. S. Bailey, P. A. Hill, and J. F. Seager, *J.C.S. Perkin I*, 1974, 967.

with the ion (VII). Further, compound (IV) behaves differently from (I) on melting, forming a tar; t.l.c. of the melt shows that it contains several components.

However, we have observed that on heating compound (IV; Z = Ts) in chlorobenzene (b.p. 132°) it rearranged to form an isomer; the product was colourless and the i.r. spectrum indicated the absence of a C=NTs group, thus eliminating structures (VIII) and (IX). The mass spectrum of the material contained no molecular ion, the base peak (m/e 206) corresponding to the loss of two TsNH₂ and one H; the ¹H n.m.r. spectrum indicated the

³ A. S. Bailey, R. Scattergood, and W. A. Warr, *J. Chem. Soc. (C)*, 1971, 2479; A. S. Bailey, A. J. Buckley, and J. F. Seager, *J.C.S. Perkin I*, 1973, 1809.

(XIV) in $\text{CF}_3\text{CO}_2\text{D}$. Similar effects were observed² in the n.m.r. spectrum of the ion (V).

The ethoxy-compound (VI; Z = Cbs) was reduced by sodium borohydride to compound (XVI). The n.m.r. spectrum of this compound in TFA contained two NH

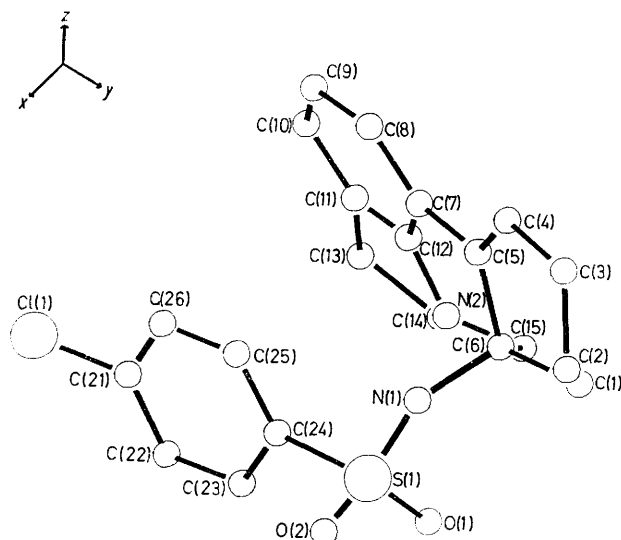


FIGURE 1 7a-p-Chlorophenylsulphonylamino-4,5,7a,8,9,10-hexahydro-7H-pyrrolo[3,2,1-de]phenanthridine, projected down (111)

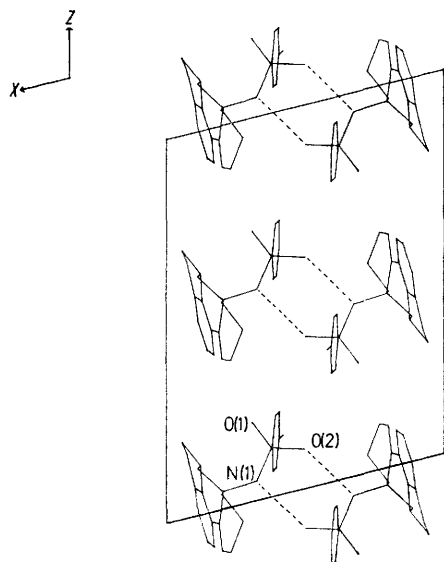


FIGURE 2 7a-p-Chlorophenylsulphonylamino-4,5,7a,8,9,10-hexahydro-7H-pyrrolo[3,2,1-de]phenanthridine; crystal structure projected down the *y*-axis

singlets (τ 1.96 and 3.56; absent in $\text{CF}_3\text{CO}_2\text{D}$) again suggesting slow exchange of the NHCbs protons, and the appearance of the signal from H(1) at high field (τ 3.45) indicated shielding of this proton by one of the CbsNH groups. Similar shielding effects have been observed⁴ in the n.m.r. spectrum of the ion (XVII). After 3 h the n.m.r. spectrum of (XVI) in TFA had changed into that of (XIV), and from the solution compound (XIV) was isolated in good yield.

Crystal Structure of 7a-p-Chlorophenylsulphonylamino-4,5,7a,8,9,10-hexahydro-7H-pyrrolo[3,2,1-de]phenanthridine.—Interatomic distances and interbond angles with standard deviations are given in Table 1. Figure 1 shows a generalised projection of the molecule, and Figure 2 a view of the crystal packing viewed down the *y*-axis.

TABLE 1

Bond lengths (Å) and bond angles (°) with estimated standard deviations

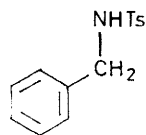
Bond lengths			
S(1)—O(1)	1.427(1)	C(6)—C(15)	1.530(3)
S(1)—O(2)	1.445(1)	C(7)—C(8)	1.404(3)
S(1)—N(1)	1.596(2)	C(7)—C(12)	1.391(3)
S(1)—C(24)	1.776(2)	C(8)—C(9)	1.385(3)
Cl(1)—C(21)	1.745(2)	C(9)—C(10)	1.392(4)
N(1)—C(6)	1.502(2)	C(19)—C(11)	1.368(3)
N(2)—C(12)	1.383(3)	C(11)—C(12)	1.391(3)
N(2)—C(14)	1.464(3)	C(11)—C(13)	1.514(3)
N(2)—C(15)	1.442(3)	C(13)—C(14)	1.527(4)
C(1)—C(2)	1.513(4)	C(21)—C(22)	1.377(3)
C(2)—C(3)	1.513(4)	C(22)—C(23)	1.382(3)
C(3)—C(4)	1.502(3)	C(23)—C(24)	1.382(3)
C(4)—C(5)	1.330(5)	C(24)—C(25)	1.390(3)
C(5)—C(6)	1.527(3)	C(25)—C(26)	1.380(3)
C(5)—C(7)	1.477(3)	C(26)—C(21)	1.380(3)
C(6)—C(1)	1.534(3)		
Bond angles			
O(1)—S(1)—O(2)	118.1(1)	C(5)—C(7)—C(12)	118.3(2)
O(1)—S(1)—N(1)	110.6(1)	C(8)—C(7)—C(12)	115.3(2)
O(2)—S(1)—N(1)	106.1(1)	C(7)—C(8)—C(9)	121.5(2)
O(1)—S(1)—C(24)	108.7(1)	C(8)—C(9)—C(10)	121.1(2)
O(2)—S(1)—C(24)	104.2(1)	C(9)—C(10)—C(11)	118.9(2)
N(1)—S(1)—C(24)	108.7(1)	C(10)—C(11)—C(12)	119.3(2)
S(1)—N(1)—C(6)	132.5(1)	C(10)—C(11)—C(13)	133.3(2)
C(12)—N(2)—C(14)	108.0(2)	C(12)—C(11)—C(13)	107.3(2)
C(12)—N(2)—C(15)	116.3(2)	C(7)—C(12)—C(11)	123.9(2)
C(14)—N(2)—C(15)	123.9(2)	C(7)—C(12)—N(2)	124.6(2)
C(6)—C(1)—C(2)	111.5(2)	C(11)—C(12)—N(2)	111.5(2)
C(1)—C(2)—C(3)	111.1(2)	C(11)—C(13)—C(14)	103.1(2)
C(2)—C(3)—C(4)	112.3(2)	C(13)—C(14)—N(2)	103.7(2)
C(3)—C(4)—C(5)	125.3(2)	N(2)—C(15)—C(6)	108.9(2)
C(4)—C(5)—C(6)	121.1(2)	Cl(1)—C(21)—C(22)	118.7(2)
C(4)—C(5)—C(7)	124.8(2)	Cl(1)—C(21)—C(26)	119.7(2)
C(6)—C(5)—C(7)	114.1(2)	C(22)—C(21)—C(26)	121.5(2)
N(1)—C(6)—C(1)	109.0(2)	C(21)—C(22)—C(23)	119.4(2)
N(1)—C(6)—C(5)	104.7(1)	C(22)—C(23)—C(24)	119.6(2)
N(1)—C(6)—C(15)	111.8(2)	S(1)—C(24)—C(23)	121.5(2)
C(1)—C(6)—C(15)	110.0(2)	S(1)—C(24)—C(25)	117.6(2)
C(15)—C(6)—C(5)	110.3(2)	C(23)—C(24)—C(25)	120.6(2)
C(5)—C(6)—C(1)	111.0(2)	C(24)—C(25)—C(26)	119.7(2)
C(5)—C(7)—C(8)	126.4(2)	C(25)—C(26)—C(21)	119.1(2)

The crystal is made up from the molecules (XIV) forming dimers at symmetry centres in which hydrogen bonds ($\text{N—H} \cdots \text{O}$, 2.935, N—H 0.84, $\text{H} \cdots \text{O}$, 2.119 Å, N—H—O 165.4°) link the oxygen atom, O(2), of one molecule to the nitrogen atom, N(1), of a second molecule. The formation of this hydrogen bond can be correlated with the lengthening of S(1)—O(2) to 1.445 Å as compared with 1.427 Å for S(1)—O(1). Similar effects have been observed in (XIX) and (XX), which also contain $\text{N—H} \cdots \text{O}$ hydrogen bonds.⁵

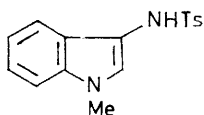
The molecule is best regarded as a 7H-pyrrolo[3,2,1-de]phenanthridine system bonded to a chlorophenylsulphonylamino-group. All the fused rings exhibit the expected conformations. The benzene ring C(7)—C(12) is planar (mean deviation 0.007, maximum 0.012 Å) and both C(5) and C(13) lie close to this plane (−0.036 and

⁵ T. S. Cameron, K. Prout, B. Denton, R. Spagna, and E. White, *J.C.S. Perkin II*, 1975, 176.

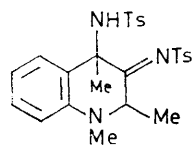
−0.037 Å, respectively) but N(2) is further out of it (−0.105 Å). This non-planarity is due to the distortion necessary to fuse the four rings. This distortion is further illustrated by the angles of the benzene ring at C(7) (115.3°) and C(12) (123.9°). The two six-membered



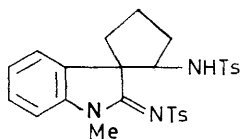
(XVIII)



(XIX)



(XX)



(XXI)

rings adopt half-chair conformations with C(5), C(7), C(12), N(2) coplanar in the first ring and C(3), C(4), C(5), C(6) coplanar in the second, as expected as a consequence of the olefinic double bond at C(4)–C(5). The olefinic double bond and its substituents lie in a plane to within ± 0.01 Å but this plane is inclined at 15.3° to the plane of the benzene ring. This inclination is about the bond C(5)–C(7), such that C(6) lies below the benzene ring, *i.e.* to the same side as the chlorophenylsulphonylamino group, and C(3) and C(4) above. The bond C(4)–C(5) has a normal double bond length (1.330 Å), and the valence-bond formulation as in (XIV) predominates. The pyrrolidine ring has an envelope-type conformation with four atoms co-planar and C(14) 0.387 Å out of plane.

The carbon atoms of the chlorobenzene ring are also strictly coplanar with the chlorine in the plane of the ring. The sulphur atom, S(1), however lies 0.133 Å below the ring, *i.e.* on the opposite side from the fused ring system. The dimensions of the chlorobenzene group are as expected and neither oxygen atom of the sulphonyl group lies in the plane of the ring as was observed in (XXI).⁶

The bond S(1)–N(1) is short (1.596 Å). This is significantly longer than 1.553 Å in (XXI) where electron delocalisation has been postulated to account for the shortening, but it is shorter than in equivalent structures where no such delocalisation is possible, *e.g.* 1.619 Å in (XVIII), 1.630 Å in (XIX), and 1.619 Å in (XXI).^{5,6} This is apparently due to increased p_π – d_π bonding. The angle between the C(6)–N(1)–S(1) and O(1)–S(1)–O(2) planes (52.6°) is suitable for such an interaction, as is the angle at N(1) (132.5°).

EXPERIMENTAL

General details and instruments used have been reported.³ U.v. spectra were determined for solutions in ethanol and n.m.r. spectra for solutions in CDCl_3 unless otherwise stated; i.r. spectra were recorded for Nujol mulls.

4,5,7a,8,9,10,11,11a-Octahydro-7a,11a-bis-(*p*-tolylsulphonylamino)pyrrolo[3,2,1-de]phenanthridinium Trifluoroacetate.—A solution of compound (IV; Z = Ts) (0.4 g)² in TFA (4 ml) was kept at 20°C for 7 days. The solvent was removed and the residue recrystallised from ethyl acetate yielding the trifluoroacetate (0.23 g), m.p. 152 – 153° (Found: C, 50.5; H, 4.2; N, 5.6. $\text{C}_{33}\text{H}_{32}\text{F}_6\text{N}_3\text{O}_8\text{S}_2$ requires C, 51.0; H, 4.1; N, 5.4%; λ_{max} , 200, 215sh, 230, and 312 nm (ϵ 39 000, 31 000, 24 900, and 4 600); ν_{max} , 1 790 (C=O) and 3 280 cm^{-1}).

4,5,7a,8,9,10-Hexahydro-7,7a-bis-(*p*-tolylsulphonylamino)-7H-pyrrolo[3,2,1-de]phenanthridine (XI; Z = Ts).—A solution of compound (IV; Z = Ts) (2.5 g) in chlorobenzene (5 ml) was boiled for 30 min (oil-bath), then cooled, and methanol (5 ml) was added. The solid was collected and recrystallised from acetonitrile; compound (XI; Z = Ts) formed small prisms, m.p. 212 – 213° (1.6 g) (Found: C, 63.7; H, 5.4; N, 7.6. $\text{C}_{29}\text{H}_{31}\text{N}_3\text{O}_4\text{S}_2$ requires C, 63.4; H, 5.6; N, 7.7%; λ_{max} , 231, 263sh, and 345 nm (ϵ 34 000, 14 000, and 4 600); ν_{max} , 1 600 (C=C) and 3 280 (NH) cm^{-1} ; τ 2.35 (2 H, d, J 9 Hz, low-field half of Ts signal), 2.7–3.4 (9 H, m, ArH), 3.65 [1 H, t (poorly resolved), J 4 Hz], 5.05–5.15 (1 H, m, collapsed to singlet on adding D_2O), 5.30 and 5.50 (each 1 H, s, NH, exchanged with D_2O), 7.1–7.4 (4 H, m), 7.60 (2 \times 3 H, s, CMe), 7.8–8.0 (3 H, m), and 8.3–8.7 (3 H, m); *m/e* (M^+ not detected) 223 (M – Ts – TsNH_2 , 30%) and 206 (M – 2TsNH_2 – H, 100%). Compound (IV; Z = Cbs) (1 g) was boiled (15 min) in chlorobenzene (2 ml) affording 7,7a-bis-(*p*-chlorophenylsulphonylamino)-4,5,7a,8,9,10-hexahydro-7H-pyrrolo[3,2,1-de]phenanthridine (XI; Z = Cbs) (62%). The same compound (m.p., t.l.c., and i.r. spectrum) was obtained by boiling (15 min) compound (VI; Z = Cbs)² in chlorobenzene. It formed small prisms (from acetonitrile), m.p. 216 – 218° (decomp.) (Found: C, 55.1; H, 4.3; N, 7.0. $\text{C}_{27}\text{H}_{25}\text{Cl}_2\text{N}_3\text{O}_4\text{S}_2$ requires C, 55.0; H, 4.2; N, 7.1%; λ_{max} , 234 and 343 nm (ϵ 40 600 and 4 800); ν_{max} , 1 590 and 3 280 cm^{-1} ; *m/e* 223 (19%) and 206 (100%) [weak peaks (<1%) at 414 and 398]. The n.m.r. spectrum of compound (XI; Z = Cbs) in TFA was recorded 1 h after making up the solution, the spectrum showing the presence of the ion (XIII) and CbsNH_2 , τ 0.98 [1 H, s, C(7)H], 2.0–2.5 (12 H, m), 3.07 [1 H, m, C(11)H], 4.60 [2 H, t, J 8 Hz, C(5)H₂], 6.08 [2 H, t, J 8 Hz, C(4)H₂], 6.6–6.7 (2 H, m), and 7.5–7.8 (2 H, m); when the spectrum was recorded as quickly as possible after making up the solution all the above peaks were observed plus signals at 1.55 (s), 3.30 (t, J 3 Hz), 5.10 (t, J 8 Hz), and 6.42 (t, J 8 Hz) associated with ion (XII). *p*-Chlorobenzenesulphonamide was the only crystalline material isolated from the TFA solution.

7a-*p*-Chlorophenylsulphonylamino-4,5,7a,8,9,10-hexahydro-7H-pyrrolo[3,2,1-de]phenanthridine (XIV).—(a) Sodium borohydride (0.4 g) was added to a suspension of compound (XI; Z = Cbs) (0.4 g) in ethanol (30 ml) and the mixture boiled (30 min). Next day the solution was neutralised (HOAc) and then poured into water (250 ml). The solid was collected and recrystallised from ethanol (yield 0.23 g, 84%); the compound formed prisms, m.p. 147 – 150° (decomp.) (Found: C, 63.2; H, 5.3; N, 7.0. $\text{C}_{21}\text{H}_{21}\text{ClN}_2\text{O}_2\text{S}$ requires C, 63.1; H, 5.3; N, 7.0%; λ_{max} , 235, 270sh, and 345 nm (ϵ 26 200, 9 600, and 5 200); ν_{max} , 1 600 and 3 260 cm^{-1} ; τ 2.30 (2 H, d, J 9 Hz, low-field half of Cbs signal), 2.65 (2 H, d, J 9 Hz, high-field half of Cbs signal), 2.9–3.5 (3 H, m, ArH), 3.80 [1 H, t, J 2 Hz, C(11)H], 5.20 (1 H, s, NH, exchanged in D_2O), 6.47 (1 H, d, J 11 Hz), and 7.05–8.75 (11 H, m);

⁶ I. J. Tickle and C. K. Prout, *J. Chem. Soc. (C)*, 1971, 3401.

m/e 400 (*M*, 17%), 225 (*M* - Cbs, 100%), 208 (16%), and 197 (225 - C₂H₄, 21%; *m** 172.1).

(b) A solution of compound (XVI) (see later) (0.25 g) in TFA (5 ml) was kept at room temperature for 20 h. The solvent was removed *in vacuo*, the residue triturated with methanol, and the solid collected. Recrystallisation from ethanol afforded (XIV) (identical in m.p., t.l.c., and i.r. spectrum) (yield 74%).

7a, 11a-Bis-(*p*-chlorophenylsulphonylamino)-4,5,7a,8,9,10, 11,11a-octahydro-7H-pyrrolo[3,2,1-de]phenanthridine (XVI).—Sodium borohydride (0.5 g) and compound (VI; Z = Cbs)² (0.5 g) were boiled (1 h) in ethanol (50 ml). Next day the product was isolated (see previous reaction) and recrystallised from chloroform-methanol (1:3). The compound formed rods (0.13 g), m.p. 166–168° (Found: C, 54.4; H, 4.8; N, 7.2; S, 10.8. C₂₇H₂₇Cl₂N₃O₄S₂ requires C, 54.7; H, 4.6; N, 7.1; S, 10.8%); λ_{max} 235, 261sh, and 318 nm (ε 31 000, 9 800, and 3 500); ν_{max} 3 300 and 3 360 cm⁻¹; τ 2.04 (2 H, d, *J* 9 Hz), 2.45–2.75 (6 H, m, ArH), 3.15 (1 H, d, *J* 9 Hz, ArH), 3.46–3.62 (2 H, m, ArH), 3.70 and 4.52 (each 1 H, s, NH, exchanged in D₂O), 6.5–7.3 (6 H, m), 7.6–7.8br (2 H, s), 8.0–8.2 (1 H, m), and 8.4–9.0 (5 H, m); *m/e* 400 (*M* - CbsNH₂, 10%), 225 (*M* - Cbs - CbsNH₂, 41%), 208 (*M* - 2CbsNH₂ - H, 10%), and 111 (C₆H₄Cl, 100%).

Crystallographic Data.—*Crystal data.* C₂₁H₂₁ClN₂O₂S, *M* = 400.9. Monoclinic, *a* = 11.93 ± 0.02, *b* = 10.088 ± 0.005, *c* = 16.23 ± 0.03 Å, β = 104.27 ± 0.09°, *U* = 1 892.05 Å³, *D_m* = 1.395, *Z* = 4, *D_c* = 1.407 g cm⁻³. Space group *P*2₁/c(*C*_{2h}⁵, No. 14, second setting). Mo-*K*_α radiation, λ = 0.710 7 Å; μ = 3.31 cm⁻¹. 2 789 Independent reflections; four-circle diffractometer.

Preliminary unit cell dimensions were obtained from oscillation, Weissenberg, and precession photographs of crystals mounted about the *b*- and *c*-axes. A diamond-shaped crystal (0.55 × 0.75 × 0.30 mm) was mounted about the *b*-axis on a Hilger and Watts four-circle diffractometer and accurate cell dimensions and orientation matrix were obtained by a least-squares fit to the accurately determined setting angles of 21 reflections.⁷ The intensities of one equivalent of each independent reflection with 2θ < 50° were measured with an ω/2θ scan and ordinate analysis⁸ with 60 steps of 0.02° counting for 1 s at each step. Reflections with *I* < 3σ_{*I*}, where σ_{*I*} is the standard deviation of the intensity *I* based on counting statistics, or whose apparent centre was more than 0.2° from the predicted position, were not included in subsequent calculations, which were based on the remaining 2 789 reflections. Lorentz and polarisation corrections were applied but no correction was made for absorption.

The structure was solved by direct methods using weighted multi-solution tangent refinement.⁹ The phases of 605 reflections with *E* > 1.2 were developed in ten cycles of refinement using phases in Table 2 to define the origin, and

TABLE 2

<i>h</i>	<i>k</i>	<i>l</i>	<i>E</i>	(°)
3	7	6	4.31	0
0	1	13	3.14	0
2	2	7	3.28	0

(-6, 1, 2) and (-3, 1, 15) as multi-solution phases. An *E* map calculated from the phase set with best figure of merit revealed all non-hydrogen atoms. This model with individual

⁷ M. Dobler and B. Duerr, personal communication.

⁸ H. C. Watson, D. M. Shotten, J. M. Cole, and H. Muirhead, *Nature*, 1970, **225**, 806.

isotropic temperature factors was refined by full matrix least-squares to *R* 0.139. The least-squares matrix was then divided into two blocks, one for positional parameters the other for the anisotropic thermal parameters and the scale factor. The refinement continued to *R* 0.095 when a difference Fourier map revealed 18 of the 21 hydrogen atoms. These were included in the refinement with isotropic temperature factors and a further least-squares cycle was calculated with Waser-type constraints¹⁰ applied so that C-H distances were 1.00 Å with an estimated standard deviation of 0.01 Å and the difference in mean square displacement along a bond X-H was 0.00 Å² with an e.s.d. of 0.01 Å². A

TABLE 3

Fractional atomic co-ordinates with standard deviations (of the last digit) in parentheses

Atom	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>
S(1)	0.378 23(4)	0.000 67(5)	0.372 68(3)
Cl(1)	0.420 50(7)	-0.615 25(6)	0.346 18(5)
O(1)	0.308 4(1)	0.051 6(2)	0.294 75(9)
O(2)	0.498 6(1)	0.038 8(1)	0.397 13(9)
N(1)	0.325 1(1)	0.040 4(2)	0.450 4(1)
N(2)	0.122 9(1)	-0.110 0(2)	0.370 8(1)
C(1)	0.200 1(2)	0.231 4(2)	0.462 2(2)
C(2)	0.275 3(2)	0.280 5(2)	0.545 8(2)
C(3)	0.231 9(2)	0.225 7(3)	0.619 1(2)
C(4)	0.200 8(2)	0.081 4(2)	0.607 7(1)
C(5)	0.188 8(2)	0.013 5(2)	0.535 9(1)
C(6)	0.205 5(2)	0.080 0(2)	0.455 2(1)
C(7)	0.162 4(2)	-0.129 6(2)	0.526 2(1)
C(8)	0.171 2(2)	-0.221 9(2)	0.592 3(1)
C(9)	0.148 3(2)	-0.355 1(2)	0.575 7(2)
C(10)	0.113 9(2)	-0.402 3(2)	0.492 7(2)
C(11)	0.103 1(2)	-0.314 7(2)	0.426 8(1)
C(12)	0.129 3(2)	-0.181 8(2)	0.444 4(1)
C(13)	0.073 3(2)	-0.330 6(3)	0.331 1(2)
C(14)	0.056 5(2)	-0.187 9(3)	0.299 2(1)
C(15)	0.113 5(2)	0.031 9(2)	0.377 7(1)
C(21)	0.406 1(2)	-0.443 8(2)	0.353 2(1)
C(22)	0.396 0(2)	-0.390 0(2)	0.429 0(1)
C(23)	0.385 3(2)	-0.254 3(2)	0.435 7(1)
C(24)	0.381 7(2)	-0.174 9(2)	0.365 8(1)
C(25)	0.392 1(2)	-0.230 0(2)	0.289 5(1)
C(26)	0.405 7(2)	-0.365 3(2)	0.283 5(1)
H(100)	0.374(2)	0.032(2)	0.497(1)
H(11)	0.118(1)	0.254(2)	0.459(2)
H(12)	0.226(2)	0.271(3)	0.415(1)
H(21)	0.357(1)	0.250(3)	0.552(2)
H(22)	0.276(2)	0.379(1)	0.551(2)
H(31)	0.163(2)	0.275(2)	0.629(2)
H(32)	0.298(2)	0.234(3)	0.671(1)
H(41)	0.190(2)	0.035(2)	0.659(1)
H(81)	0.197(2)	-0.191(2)	0.651 7(8)
H(91)	0.160(2)	-0.416(2)	0.625(1)
H(101)	0.096(2)	-0.496(1)	0.476(2)
H(131)	0.005(2)	-0.391(2)	0.314(2)
H(132)	0.136(2)	-0.377(3)	0.312(2)
H(141)	-0.027(1)	-0.163(3)	0.287(2)
H(142)	0.090(2)	-0.164(3)	0.251(1)
H(151)	0.124(2)	0.074(2)	0.325(1)
H(152)	0.035(1)	0.054(2)	0.385(1)
H(221)	0.397(2)	-0.448(2)	0.479(1)
H(231)	0.379(2)	-0.318(2)	0.491 1(9)
H(251)	0.393(2)	-0.169(2)	0.242(1)
H(261)	0.417(2)	-0.410(2)	0.232(1)

second difference map showed diffuse electron density close to positions calculated as appropriate for the remaining three hydrogen atoms. Further refinement converged at *R* 0.036. In the latter stages of refinement a weighting scheme of the form $w = [1 + (F_o/32 - 1)^2]^{-1}$ was applied

⁹ G. Germain, P. Main, and M. M. Woolfson, *Acta Cryst.*, 1971, **A27**, 368.

¹⁰ J. Waser, *Acta Cryst.*, 1963, **16**, 1091.

to each reflection and anomalous dispersion effects of S and Cl were allowed for.¹¹ Final atomic parameters are given in Table 3, and thermal parameters and observed and calculated structure factor amplitudes are listed in Supplementary Publication No. SUP 21848 (26 pp., 1 microfiche).†

All calculations were carried out on the Oxford University

† For details of Supplementary Publications see Notice to Authors No. 7 in *J.C.S. Perkin I*, 1975, Index issue.

Computing Laboratory's ICL 1906A computer using the CRYSTALS program system.¹² Atomic scattering factors were taken from ref. 13.

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¹¹ D. T. Cromer and J. T. Waber, *J. Chem. Phys.*, 1970, **53**, 1891.

¹² J. R. Carruthers, personal communication.

¹³ D. T. Cromer and J. B. Mann, *Acta Cryst.*, 1968, **A24**, 321.
