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The Structures of Four Reaction Products of 1,2,2a,3,4,5-Hexahydropyrrolo[3,2,1-*jk*]carbazole with *p*-Chlorobenzenesulphonyl Azide

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(*Received* 24 *November* 1979; *accepted* 18 *February* 1980)

Abstract

The crystal structures of 12-(*p*-chlorophenylsulphonylamino)-6,7,10,11-tetrahydroazocino[1,2-*a*]indol-8(9*H*)-one, $C_{20}H_{19}ClN_2O_3S$ (I) [monoclinic, a = 10·404 (3), b = 9·091 (4), c = 10·213 (1) Å, β = 102·48 (2)°, Z = 2, space group $P2_1$, 2050 reflexions, R_w = 0·043], 14-(*p*-chlorophenyl)-8-(*p*-chlorophenylsulphonylamino)-13-oxa-14-thia-1,15-diazapentacyclo[10.4.2.$0^{2,7}$.$0^{8,16}$.$0^{12,16}$]octadeca-2,4,6,14-tetraene *S*-oxide, $C_{26}H_{23}Cl_2N_3O_4S_2$ (II) [triclinic, a = 8·127 (2), b = 12·476 (4), c = 12·732 (5) Å, α = 100·73 (3), β = 90·77 (3), γ = 94·93 (3)°, Z = 2, space group $P\bar{1}$, 2378 reflexions, R_w = 0·054], 6-(*p*-chlorophenylsulphonylamino)-2,2a,3,4,5,6-hexahydro-2a,6-methano-1*H*-azeto[1,2-*a*][1]benzazocin-12-one, $C_{20}H_{19}ClN_2O_3S$ (III) [triclinic, a = 6·781 (2), b = 9·781 (2), c = 14·151 (2) Å, α = 98·88 (2), β = 97·85 (2), γ = 96·74 (2)°, Z = 2, space group $P\bar{1}$, 2386 reflexions, R_w = 0·044], and 8-(*p*-chlorophenylsulphonylamino)-4-hydroxyimino-1,2,3,4,5,6,7,8-octahydro-1,8-methano[1]benzazecin-13-one pyridine solvate, $C_{20}H_{20}ClN_3O_4S.C_5H_5N$ (IV) [triclinic, a = 9·981 (2), b = 11·624 (2), c = 13·088 (2) Å, α = 65·46 (1), β = 71·74 (1), γ = 63·34 (2)°, Z = 2, space group $P\bar{1}$, 2112 reflexions, R_w = 0·057] have been determined.

Introduction

p-Chlorobenzenesulphonyl azide reacts with 1,2,2a,3,4,5-hexahydropyrrolo[3,2,1-*jk*]carbazole in CCl_4 in accordance with the scheme on page 1847 (Bailey, Scott & Vandrevala, 1980). The structures of the products (I–IV) were determined by X-ray methods.

Experimental

Transparent plate-like crystals of all four compounds were prepared. After survey photography by Weissenberg and precession techniques, the crystals were set up on a Nonius CAD-4F, PDP8-controlled κ-geometry diffractometer which used Mo $K\alpha$ radiation from a graphite monochromator. Cell dimensions and the orientation matrix were obtained by least squares from the setting angles of 25 reflexions.

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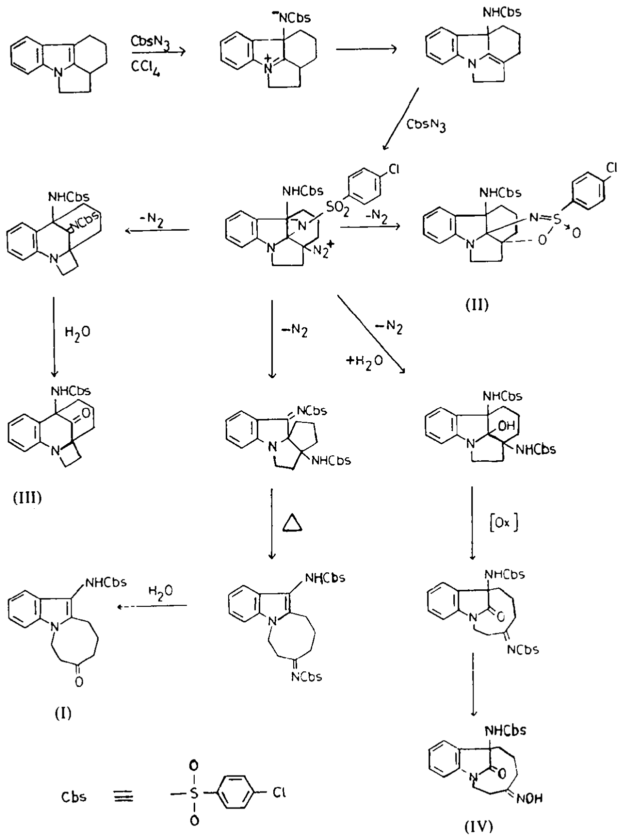

Crystal data

(*a*) Compound (I): $C_{20}H_{19}ClN_2O_3S$, M_r = 402·9, monoclinic, a = 10·404 (3), b = 9·091 (4), c = 10·213 (1) Å, β = 102·48 (2)°, U = 943·1 Å^3. Systematic extinctions: $0k0$, $k = 2n + 1$. Space group $P2_1$ or $P2_1/m$; $P2_1$ from structure analysis. D_c = 1·42 for Z = 2, D_m = 1·42 Mg m^{-3}. Mo $K\alpha$ radiation, λ = 0·71069 Å, μ = 0·33 mm^{-1}.

(*b*) Compound (II): $C_{26}H_{23}Cl_2N_3O_4S_2$, M_r = 576·5, triclinic, a = 8·127 (2), b = 12·476 (4), c = 12·732 (5) Å, α = 100·73 (3), β = 90·77 (3), γ = 94·93 (3)°, U = 1263·0 Å^3. Space group $P\bar{1}$ or $P1$; $P\bar{1}$ from structure analysis. D_c = 1·52 for Z = 2, D_m = 1·50 Mg m^{-3}. Mo $K\alpha$ radiation, λ = 0·71069 Å, μ = 0·45 mm^{-1}.

(*c*) Compound (III): $C_{20}H_{19}ClN_2O_3S$, M_r = 402·9, triclinic, a = 6·781 (2), b = 9·781 (2), c = 14·151 (2) Å, α = 98·88 (2), β = 97·85 (2), γ = 96·74 (2)°, U = 909·4 Å^3. Space group $P\bar{1}$ or $P1$; $P\bar{1}$ from structure analysis. D_c = 1·47 for Z = 2, D_m = 1·49 Mg m^{-3}. Mo $K\alpha$ radiation, λ = 0·71069 Å, μ = 0·34 mm^{-1}.

(*d*) Compound (IV): $C_{20}H_{20}ClN_3O_4S.C_5H_5N$, M_r = 513·0, triclinic, a = 9·981 (2), b = 11·624 (2), c = 13·088 (2) Å, α = 65·46 (1), β = 71·74 (1), γ = 63·34 (2)°, U = 1219·3 Å^3. Space group $P\bar{1}$ or $P1$; $P\bar{1}$ from structure analysis. D_c = 1·40 for Z = 2, D_m = 1·38 Mg m^{-3}. Mo $K\alpha$ radiation, λ = 0·71069 Å, μ = 0·27 mm^{-1}.

For each crystal, the intensities of every independent reflexion with $\sin\theta/\lambda$ < 0·66 (I), 0·59 (II), 0·70 (III), 0·59 (IV) Å^{-1} were measured with an $\omega/2\theta$ scan, a variable scan rate and an ω scan angle of (1·00 + 0·35 tan θ)°. Reflexions with $I < 3\sigma(I)$, where $\sigma(I)$ is the e.s.d. based on simple counting statistics, were not included in the subsequent calculations. (I), (II), (III) and (IV) gave 2050, 2378, 2386 and 2112 independent structure amplitudes corrected for Lorentz and polarization effects, but not absorption.

Structure solution and refinement

(*a*) *Compound* (I)

The ten non-hydrogen atoms of the *p*-chlorophenylsulphonyl residue, together with the N atom attached to S, were located from an E map based on the best phase set after weighted, multi-solution tangent refinement (Germain, Main & Woolfson, 1971), with three origin-defining phases ($\underline{2}10$, $30\bar{4}$, $80\bar{1}$) and two multi-solution phases (471, $11\bar{5}$); 245 reflexions with $E > 1{\cdot}4$ were used in this refinement. Two subsequent difference syntheses revealed the remaining 16 non-hydrogen atoms. During refinement the sum of the coordinates along the polar axis was set to zero to determine the origin (Carruthers, 1975). Refinement was by least squares with a large-block approximation to the normal matrix: a 2 × 2 block was calculated from the derivatives of the scale and dummy overall isotropic temperature factor (Rollett, 1965), one from those of the positional parameters and one from those of the corresponding temperature factors. All non-hydrogen atoms had anisotropic temperature factors. The H atoms were located from difference syntheses, and were included in the refinement with isotropic temperature factors and Waser-type constraints (Waser, 1963; Rollett, 1969) applied to the bond lengths and angles involving the H atoms. In the final stages of refinement, each reflexion was assigned a weight $w = 1/\sum_{r=1}^{n} A_r T_r^{*}(X)$, where n is the number of coefficients, A_r, for a Chebyshev series, T_r^{*} is the polynomial function and X is $|F_o|/|F_o(\max)|$. Three coefficients, A_r, were used with values 44·40, 59·08 and 15·19 (Rollett, 1965), and the final R_w was 0·043 for 2050 reflexions.

(*b*) *Compound* (II)

With *MULTAN* 77 (Main, Lessinger, Woolfson, Germain & Declercq, 1977), 36 of the 37 non-hydrogen atoms were located from an E map based on the best phase set after weighted, multi-solution tangent refinement with three origin-defining phases ($1\underline{5}2$, $3\bar{4}3$, $01\bar{6}$) and three multi-solution phases ($2\bar{2}0$, $25\bar{9}$, $48\bar{7}$); 250 phases with normalized structure factors >1·50 were used in this refinement. Two cycles of refinement of the atomic coordinates and isotropic temperature factors, followed by a difference synthesis, revealed the last atom. Refinement was by least squares with a large-block approximation to the normal matrix: a 3 × 3 block was calculated from the derivatives of the scale, dummy overall isotropic temperature factor and overall isotropic extinction parameter (Larson, 1967), one from those of the positional parameters, one from those of the U_{ii} components of the thermal parameters of the non-hydrogen atoms and the H atom isotropic thermal parameters, and one from the U_{ij} components of the thermal parameters of the non-hydrogen atoms. All non-hydrogen atoms had anisotropic temperature factors. The H atoms were located from difference syntheses and were included in the refinement with isotropic temperature factors and Waser-type constraints applied to the bond lengths and angles involving H atoms. In the final stages of refinement the overall isotropic extinction parameter (165·3) was introduced. A Chebyshev-type weighting scheme was used with three coefficients, A_r, with values 15·59, 21·09 and 6·04, and the final R_w was 0·054 for 2378 reflexions.

(*c*) *Compound* (III)

The ten non-hydrogen atoms of the *p*-chlorophenylsulphonyl residue, together with the N atom attached to S, were located from an *E* map based on the best phase set after weighted, multi-solution tangent refinement, with three origin-defining phases ($12\bar{2}$, $22\bar{5}$, $3\bar{9}1$) and three multi-solution phases ($37\bar{2}$, $3\bar{2}2$, $2\bar{3}\bar{3}$); 214 reflexions with $E > 1{\cdot}5$ were used in this refinement. Subsequent difference syntheses revealed the remaining 16 non-hydrogen atoms. Refinement was by least squares with a large-block approximation to the normal matrix: one block was calculated from the derivatives of the positional parameters, and one from those of the scale, overall isotropic extinction parameter and corresponding temperature factors. All non-hydrogen atoms had anisotropic temperature factors. The H atoms were located from difference syntheses and were included in the refinement with isotropic temperature factors and Waser-type constraints applied to the bond lengths and angles involving H atoms. In the final stages of refinement the overall isotropic extinction parameter (84·5) was introduced. A Chebyshev-type weighting scheme was used with three coefficients, A_r, with values 5·68, 7·97 and 2·67 and the final R_w was 0·044 for 2386 reflexions.

(*d*) *Compound* (IV)

With *MULTAN* 1977, all the non-hydrogen atoms were located from an *E* map based on the best phase set after weighted, multi-solution tangent refinement with three origin-defining phases ($20\bar{3}$, $2\bar{1}5$, 3,10,6) and five multi-solution phases (384, 036, $4\bar{5}2$, $1\bar{4}\bar{1}$, 7,10,1); 300 phases with normalized structure factors >1·38 were used in this refinement. Refinement was by least squares with a large-block approximation to the normal matrix: a 2 × 2 block was calculated from the derivatives of the scale and dummy overall isotropic temperature factor, one from those of the positional parameters, one from those of the U_{ii} components of the thermal parameters of the non-hydrogen atoms, and one from those of the U_{ij} components of the thermal parameters of the non-hydrogen atoms. All non-hydrogen atoms had anisotropic temperature factors. The H atoms were located from difference syntheses. They were positioned geometrically

Table 1. *Atomic parameters for compound* (I)

	x	*y*	*z*
Cl(1)	0·8338 (1)	0·3276 (4)	0·83123 (8)
S(1)	0·91452 (5)	0·2479 (4)	0·24607 (7)
N(1)	0·7817 (2)	0·1725 (4)	0·1578 (2)
N(2)	0·7231 (2)	−0·2177 (4)	0·1250 (2)
O(1)	0·9159 (2)	0·3942 (4)	0·1905 (2)
O(2)	1·0225 (2)	0·1513 (4)	0·2489 (2)
O(3)	0·4661 (2)	−0·1703 (4)	−0·1587 (3)
C(1)	0·7150 (3)	−0·3579 (5)	0·0556 (3)
C(2)	0·5851 (3)	−0·3740 (5)	−0·0478 (3)
C(3)	0·5650 (2)	−0·2445 (5)	−0·1426 (3)
C(4)	0·6719 (3)	−0·2100 (5)	−0·2160 (3)
C(5)	0·7457 (3)	−0·0667 (5)	−0·1668 (3)
C(6)	0·8403 (2)	−0·0764 (5)	−0·0285 (3)
C(7)	0·7744 (2)	−0·0885 (4)	0·0880 (2)
C(8)	0·7515 (2)	0·0212 (4)	0·1722 (2)
C(9)	0·6808 (2)	−0·0399 (4)	0·2649 (2)
C(10)	0·6269 (3)	0·0165 (5)	0·3690 (3)
C(11)	0·5540 (3)	−0·0760 (5)	0·4319 (3)
C(12)	0·5351 (4)	−0·2250 (5)	0·3944 (3)
C(13)	0·5888 (3)	−0·2838 (5)	0·2938 (3)
C(14)	0·6620 (2)	−0·1911 (4)	0·2306 (2)
C(15)	0·8934 (2)	0·2702 (4)	0·4124 (2)
C(16)	0·9580 (3)	0·1796 (5)	0·5141 (3)
C(17)	0·9398 (3)	0·1982 (5)	0·6439 (3)
C(18)	0·8570 (3)	0·3062 (5)	0·6694 (3)
C(19)	0·7918 (3)	0·3988 (5)	0·5690 (3)
C(20)	0·8095 (3)	0·3801 (5)	0·4395 (3)
H(1)	0·713 (3)	0·230 (2)	0·143 (4)
H(101)	0·789 (3)	−0·366 (3)	0·010 (2)
H(102)	0·724 (3)	−0·443 (3)	0·125 (3)
H(201)	0·511 (3)	−0·375 (3)	0·001 (2)
H(202)	0·590 (3)	−0·468 (3)	−0·103 (2)
H(401)	0·633 (2)	−0·200 (3)	−0·310 (3)
H(402)	0·737 (3)	−0·294 (3)	−0·204 (3)
H(501)	0·794 (2)	−0·034 (3)	−0·233 (2)
H(502)	0·683 (3)	0·011 (4)	−0·166 (2)
H(601)	0·900 (3)	−0·161 (4)	−0·0274 (8)
H(602)	0·895 (3)	0·004 (4)	−0·0166 (8)
H(1001)	0·639 (3)	0·127 (4)	0·396 (3)
H(1101)	0·512 (4)	−0·036 (2)	0·505 (4)
H(1201)	0·484 (3)	−0·290 (2)	0·444 (3)
H(1301)	0·571 (3)	−0·381 (4)	0·265 (3)
H(1601)	1·018 (4)	0·106 (4)	0·496 (2)
H(1701)	0·985 (3)	0·132 (4)	0·717 (3)
H(1901)	0·729 (4)	0·479 (4)	0·590 (1)
H(2001)	0·764 (3)	0·439 (4)	0·370 (3)

Table 2. *Atomic parameters for compound* (II)

	x	*y*	*z*
Cl(1)	0·2535 (2)	0·5209 (1)	0·0633 (1)
Cl(2)	0·9062 (2)	1·2389 (1)	1·0400 (1)
S(1)	0·6639 (1)	0·91096 (8)	0·35781 (8)
S(2)	0·6516 (1)	0·79173 (8)	0·75786 (8)
O(1)	0·5627 (4)	0·9997 (2)	0·3860 (2)
O(2)	0·8155 (3)	0·9299 (2)	0·3050 (2)
O(3)	0·5201 (4)	0·7446 (2)	0·8122 (2)
O(4)	0·8089 (3)	0·7256 (2)	0·7625 (2)
N(1)	0·7006 (4)	0·8683 (2)	0·4660 (2)
N(2)	0·6358 (4)	0·7975 (2)	0·6409 (2)
N(3)	0·6678 (4)	0·6226 (2)	0·5223 (3)
C(1)	0·8849 (5)	0·6842 (3)	0·2917 (3)
C(2)	0·8588 (6)	0·5865 (4)	0·2177 (3)
C(3)	0·7665 (6)	0·4970 (3)	0·2448 (4)
C(4)	0·7043 (5)	0·5026 (3)	0·3442 (4)
C(5)	0·7276 (4)	0·6005 (3)	0·4182 (3)
C(6)	0·8156 (5)	0·6910 (3)	0·3908 (3)
C(7)	0·8201 (4)	0·7881 (3)	0·4836 (3)
C(8)	0·7493 (4)	0·7308 (3)	0·5762 (3)
C(9)	0·8831 (5)	0·7010 (3)	0·6562 (3)
C(10)	1·0525 (5)	0·7630 (3)	0·6636 (3)
C(11)	1·1112 (5)	0·7800 (3)	0·5549 (3)
C(12)	0·9926 (5)	0·8477 (3)	0·5080 (3)
C(13)	0·8795 (5)	0·5770 (3)	0·6282 (4)
C(14)	0·7031 (5)	0·5420 (3)	0·5894 (4)
C(15)	0·7131 (5)	0·9215 (3)	0·8356 (3)
C(16)	0·7846 (7)	1·0026 (4)	0·7914 (3)
C(17)	0·8415 (7)	1·1002 (4)	0·8537 (4)
C(18)	0·8295 (6)	1·1165 (4)	0·9607 (4)
C(19)	0·754 (1)	1·0365 (5)	1·0061 (4)
C(20)	0·6945 (9)	0·9383 (5)	0·9440 (4)
C(21)	0·5444 (5)	0·8032 (3)	0·2724 (3)
C(22)	0·4163 (5)	0·7458 (3)	0·3135 (3)
C(23)	0·3255 (5)	0·6604 (4)	0·2486 (3)
C(24)	0·3625 (6)	0·6333 (4)	0·1430 (4)
C(25)	0·4855 (6)	0·6912 (4)	0·1004 (3)
C(26)	0·5781 (5)	0·7764 (4)	0·1653 (3)
H(11)	0·634 (4)	0·888 (3)	0·519 (2)
H(1)	0·954 (6)	0·747 (3)	0·274 (2)
H(2)	0·900 (5)	0·582 (1)	0·145 (3)
H(3)	0·749 (4)	0·429 (3)	0·193 (2)
H(4)	0·648 (6)	0·438 (3)	0·364 (2)
H(101)	1·050 (2)	0·834 (3)	0·711 (2)
H(102)	1·135 (3)	0·721 (2)	0·698 (2)
H(111)	1·114 (2)	0·708 (3)	0·507 (2)
H(112)	1·221 (4)	0·818 (2)	0·562 (1)
H(121)	0·984 (1)	0·919 (2)	0·559 (2)
H(122)	1·036 (2)	0·867 (2)	0·445 (2)
H(131)	0·908 (2)	0·546 (1)	0·689 (2)
H(132)	0·961 (3)	0·553 (1)	0·571 (2)
H(141)	0·691 (2)	0·464 (3)	0·547 (2)
H(142)	0·626 (3)	0·544 (2)	0·650 (2)
H(16)	0·785 (4)	0·995 (1)	0·715 (3)
H(17)	0·900 (5)	1·153 (2)	0·823 (1)
H(19)	0·728 (4)	1·052 (1)	1·081 (3)
H(20)	0·615 (4)	0·890 (2)	0·974 (1)
H(22)	0·392 (3)	0·765 (2)	0·390 (3)
H(23)	0·234 (4)	0·622 (3)	0·276 (1)
H(25)	0·513 (3)	0·669 (2)	0·028 (3)
H(26)	0·669 (5)	0·813 (3)	0·138 (1)

(N—H = 0·90, C—H = 1·0, O—H = 1·0 Å; $U_{\rm iso}$ = 0·05 Å^2) and included in the structure factor calculations, their location being readjusted after each cycle. In the final stages of refinement, a Chebyshev-

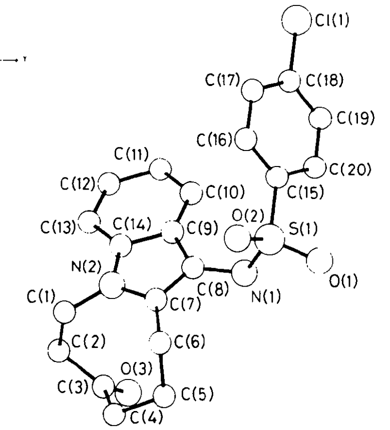

Fig. 1. The molecular structure of compound (I) seen in projection down *a*.

Table 3. *Atomic parameters for compound* (III)

	x	*y*	*z*
Cl(1)	−0·3614 (2)	0·2698 (1)	−0·45145 (9)
S(1)	0·3515 (1)	0·08583 (8)	−0·19686 (6)
O(1)	0·4395 (3)	−0·0074 (2)	−0·2609 (2)
O(2)	0·4723 (4)	0·2094 (2)	−0·1398 (2)
O(3)	0·5729 (3)	−0·1401 (2)	−0·0917 (2)
N(1)	0·2461 (4)	0·0048 (2)	−0·1197 (2)
N(2)	0·2375 (4)	−0·4197 (2)	−0·2407 (2)
C(1)	0·3943 (5)	−0·4790 (4)	−0·2896 (3)
C(2)	0·5397 (5)	−0·4418 (3)	−0·1930 (3)
C(3)	0·3773 (4)	−0·3660 (3)	−0·1492 (2)
C(4)	0·2960 (5)	−0·4116 (3)	−0·0615 (2)
C(5)	0·1038 (5)	−0·3491 (3)	−0·0459 (2)
C(6)	0·1271 (5)	−0·1901 (3)	−0·0429 (2)
C(7)	0·2129 (4)	−0·1494 (3)	−0·1324 (2)
C(8)	−0·0938 (4)	−0·1511 (3)	−0·2572 (2)
C(9)	−0·2190 (5)	−0·2057 (4)	−0·3446 (2)
C(10)	−0·1783 (5)	−0·3244 (4)	−0·4001 (3)
C(11)	−0·0234 (5)	−0·3934 (4)	−0·3681 (2)
C(12)	0·0989 (4)	−0·3408 (3)	−0·2791 (2)
C(13)	0·0698 (4)	−0·2139 (3)	−0·2258 (2)
C(14)	0·4118 (4)	−0·2091 (3)	−0·1257 (2)
C(15)	0·1526 (5)	0·1419 (3)	−0·2676 (2)
C(16)	0·0102 (5)	0·2092 (4)	−0·2244 (3)
C(17)	−0·1461 (6)	0·2513 (4)	−0·2799 (3)
C(18)	−0·1585 (5)	0·2240 (4)	−0·3794 (3)
C(19)	−0·0175 (5)	0·1612 (4)	−0·4238 (3)
C(20)	0·1395 (5)	0·1186 (4)	−0·3671 (2)
H(1)	0·298 (5)	0·043 (2)	−0·059 (2)
H(101)	0·436 (2)	−0·430 (3)	−0·342 (2)
H(102)	0·363 (2)	−0·580 (3)	−0·316 (2)
H(201)	0·666 (4)	−0·378 (3)	−0·196 (1)
H(202)	0·571 (4)	−0·523 (3)	−0·164 (1)
H(401)	0·260 (3)	−0·515 (3)	−0·076 (2)
H(402)	0·399 (3)	−0·374 (3)	−0·004 (2)
H(501)	0·066 (4)	−0·369 (2)	0·016 (2)
H(502)	−0·006 (4)	−0·395 (2)	−0·100 (2)
H(601)	−0·001 (5)	−0·157 (2)	−0·040 (2)
H(602)	0·215 (4)	−0·143 (2)	0·016 (2)
H(801)	−0·122 (4)	−0·070 (4)	−0·218 (2)
H(901)	−0·331 (5)	−0·160 (3)	−0·366 (1)
H(1001)	−0·265 (4)	−0·363 (2)	−0·459 (2)
H(1101)	0·003 (4)	−0·478 (4)	−0·408 (2)
H(1601)	0·020 (3)	0·225 (4)	−0·156 (2)
H(1701)	−0·254 (5)	0·288 (4)	−0·251 (1)
H(1901)	−0·026 (3)	0·148 (4)	−0·491 (2)
H(2001)	0·239 (5)	0·075 (4)	−0·397 (1)

Table 4. *Atomic parameters for compound* (IV)

	x	*y*	*y*
Cl(1)	0·8457 (2)	−0·0166 (1)	1·2632 (1)
S(1)	0·4287 (1)	0·3277 (1)	0·8876 (1)
O(1)	0·3680 (3)	0·4651 (3)	0·8880 (3)
O(2)	0·3292 (4)	0·2611 (4)	0·9036 (3)
O(3)	0·4151 (3)	0·6100 (3)	0·6251 (2)
O(4)	0·4676 (4)	0·8993 (3)	0·3943 (3)
N(1)	0·5420 (4)	0·3151 (3)	0·7697 (3)
N(2)	0·5945 (4)	0·6250 (3)	0·6901 (3)
N(3)	0·5481 (5)	0·7746 (4)	0·3795 (4)
C(1)	0·5772 (5)	0·7653 (4)	0·6219 (4)
C(2)	0·6877 (5)	0·7691 (5)	0·5101 (4)
C(3)	0·6597 (5)	0·7100 (5)	0·4372 (3)
C(4)	0·7636 (6)	0·5767 (5)	0·4189 (4)
C(5)	0·8194 (5)	0·4524 (5)	0·5192 (4)
C(6)	0·7150 (5)	0·3735 (5)	0·5944 (4)
C(7)	0·6261 (4)	0·4058 (4)	0·7071 (3)
C(8)	0·5269 (4)	0·5576 (4)	0·6706 (3)
C(9)	0·7156 (4)	0·5311 (4)	0·7518 (3)
C(10)	0·7334 (4)	0·3998 (4)	0·7700 (3)
C(11)	0·8452 (5)	0·2918 (4)	0·8282 (4)
C(12)	0·9377 (5)	0·3178 (5)	0·8694 (4)
C(13)	0·9194 (5)	0·4495 (5)	0·8501 (4)
C(14)	0·8079 (5)	0·5598 (5)	0·7904 (4)
C(15)	0·5433 (5)	0·2298 (4)	0·9958 (3)
C(16)	0·6043 (5)	0·0889 (4)	1·0268 (4)
C(17)	0·6940 (5)	0·0130 (4)	1·1108 (4)
C(18)	0·7262 (5)	0·0792 (4)	1·1605 (4)
C(19)	0·6672 (5)	0·2176 (4)	1·1319 (4)
C(20)	0·5738 (6)	0·2939 (4)	1·0500 (4)
N(21)	0·2060 (5)	0·0609 (4)	0·2937 (4)
C(22)	0·0948 (8)	0·0995 (6)	0·3727 (4)
C(23)	−0·0379 (8)	0·1996 (7)	0·3527 (6)
C(24)	−0·0627 (6)	0·2683 (6)	0·2475 (8)
C(25)	0·0438 (8)	0·2381 (6)	0·1612 (5)
C(26)	0·1802 (7)	0·1306 (7)	0·1873 (5)
H(1)	0·5533	0·2513	0·7424
H(4)	0·3811	0·9501	0·3501
H(101)	0·5991	0·8071	0·6649
H(102)	0·4708	0·8178	0·6052
H(201)	0·6765	0·8658	0·4656
H(202)	0·7935	0·7153	0·5278
H(401)	0·8556	0·5914	0·3656
H(402)	0·7090	0·5544	0·3817
H(501)	0·8450	0·4823	0·5697
H(502)	0·9138	0·3869	0·4885
H(601)	0·7790	0·2742	0·6151
H(602)	0·6389	0·3941	0·5484
H(11)	0·8598	0·1962	0·8408
H(12)	1·0183	0·2402	0·9134
H(13)	0·9879	0·4659	0·8798
H(14)	0·7949	0·6555	0·7757
H(16)	0·5829	0·0424	0·9878
H(17)	0·7350	−0·0887	1·1353
H(19)	0·6915	0·2631	1·1701
H(20)	0·5281	0·3953	1·0297
H(22)	0·1115	0·0496	0·4537
H(23)	−0·1182	0·2224	0·4174
H(24)	−0·1625	0·3440	0·2318
H(25)	0·0267	0·2905	0·0805
H(26)	0·2613	0·1052	0·1238

type weighting scheme was introduced with three coefficients, A_r, with values 164·66, 219·36 and 63·06 and the final R_w was 0·057 for 2112 reflexions.

Table 5. *Compound* (I) ($C_{20}H_{19}ClN_2O_3S$): *bond distances* (Å) *and angles* (°)

Cl(1)—C(18)	1·732 (3)	C(6)—C(7)	1·500 (3)
S(1)—N(1)	1·629 (2)	C(7)—C(8)	1·371 (3)
S(1)—O(1)	1·448 (2)	C(8)—C(9)	1·431 (3)
S(1)—O(2)	1·421 (2)	C(9)—C(10)	1·402 (3)
S(1)—C(15)	1·771 (3)	C(9)—C(14)	1·421 (3)
N(1)—C(8)	1·425 (3)	C(10)—C(11)	1·381 (4)
N(2)—C(1)	1·451 (3)	C(11)—C(12)	1·409 (4)
N(2)—C(7)	1·377 (3)	C(12)—C(13)	1·379 (4)
N(2)—C(14)	1·387 (3)	C(13)—C(14)	1·386 (4)
C(1)—C(2)	1·533 (4)	C(15)—C(16)	1·381 (4)
C(2)—C(3)	1·510 (4)	C(16)—C(17)	1·390 (5)
C(3)—C(4)	1·502 (4)	C(17)—C(18)	1·367 (5)
C(3)—O(3)	1·212 (4)	C(18)—C(19)	1·385 (4)
C(4)—C(5)	1·540 (4)	C(19)—C(20)	1·384 (4)
C(5)—C(6)	1·539 (4)	C(20)—C(15)	1·393 (4)

N(1)—S(1)—O(1)	104·4 (1)	C(9)—C(8)—N(1)	126·8 (2)
N(1)—S(1)—O(2)	108·9 (1)	C(7)—C(8)—C(9)	108·4 (2)
N(1)—S(1)—C(15)	108·2 (1)	C(8)—C(9)—C(10)	134·9 (2)
O(1)—S(1)—O(2)	120·1 (1)	C(8)—C(9)—C(14)	106·0 (2)
O(1)—S(1)—C(15)	106·5 (1)	C(10)—C(9)—C(14)	119·0 (2)
O(2)—S(1)—C(15)	108·2 (1)	C(9)—C(10)—C(11)	118·7 (3)
S(1)—N(1)—C(8)	121·9 (2)	C(10)—C(11)—C(12)	121·1 (3)
C(1)—N(2)—C(7)	126·9 (2)	C(11)—C(12)—C(13)	121·4 (3)
C(1)—N(2)—C(14)	123·0 (2)	C(12)—C(13)—C(14)	117·6 (3)
C(7)—N(2)—C(14)	109·5 (2)	C(13)—C(14)—N(2)	130·3 (2)
N(2)—C(1)—C(2)	111·7 (2)	C(13)—C(14)—C(9)	122·3 (2)
C(1)—C(2)—C(3)	110·3 (2)	N(2)—C(14)—C(9)	107·4 (2)
C(2)—C(3)—O(3)	121·1 (3)	S(1)—C(15)—C(16)	120·8 (2)
C(2)—C(3)—C(4)	117·9 (3)	S(1)—C(15)—C(20)	119·1 (2)
O(3)—C(3)—C(4)	121·0 (3)	C(16)—C(15)—C(20)	120·1 (3)
C(3)—C(4)—C(5)	112·6 (2)	C(15)—C(16)—C(17)	120·0 (3)
C(4)—C(5)—C(6)	115·2 (3)	C(16)—C(17)—C(18)	119·4 (3)
C(5)—C(6)—C(7)	114·9 (2)	C(17)—C(18)—C(19)	121·6 (3)
C(6)—C(7)—N(2)	123·4 (2)	C(17)—C(18)—Cl(1)	119·1 (3)
C(6)—C(7)—C(8)	128·0 (2)	C(19)—C(18)—Cl(1)	119·4 (3)
N(2)—C(7)—C(8)	108·6 (2)	C(18)—C(19)—C(20)	119·1 (3)
C(7)—C(8)—N(1)	124·4 (2)	C(19)—C(20)—C(15)	119·8 (3)

All calculations were performed on the Oxford University ICL 1906A computer and the University of London CDC 7600 computer [compound (II) only] with the *CRYSTALS* package (Carruthers, 1975). Complex neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Results and discussion

The final atomic positional parameters are given in Tables 1, 2, 3 and 4,* and bond lengths and angles, with e.s.d.'s calculated from the variance–covariance matrix in Tables 5, 6, 7 and 8. Projections of the

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* Lists of structure factors and thermal parameters for all four compounds have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35171 (95 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 6. *Compound* (II) ($C_{26}H_{23}Cl_2N_3O_4S_2$): *bond distances* (Å) *and angles* (°)

Cl(1)–C(24)	1·735 (4)	C(6)–C(7)	1·524 (5)
Cl(2)–C(18)	1·728 (5)	C(7)–C(8)	1·578 (5)
S(1)–O(1)	1·431 (3)	C(7)–C(12)	1·529 (5)
S(1)–O(2)	1·434 (3)	C(8)–C(9)	1·593 (5)
S(1)–N(1)	1·599 (3)	C(9)–C(10)	1·513 (5)
S(1)–C(21)	1·771 (4)	C(9)–C(13)	1·519 (5)
S(2)–O(3)	1·425 (3)	C(10)–C(11)	1·516 (5)
S(2)–O(4)	1·587 (3)	C(11)–C(12)	1·520 (5)
S(2)–N(2)	1·508 (3)	C(13)–C(14)	1·511 (6)
S(2)–C(15)	1·761 (4)	C(15)–C(16)	1·341 (6)
O(4)–C(9)	1·479 (4)	C(16)–C(17)	1·364 (6)
N(1)–C(7)	1·498 (4)	C(17)–C(18)	1·346 (6)
N(2)–C(8)	1·455 (4)	C(18)–C(19)	1·351 (7)
N(3)–C(5)	1·404 (5)	C(19)–C(20)	1·373 (7)
N(3)–C(8)	1·491 (4)	C(20)–C(15)	1·369 (6)
N(3)–C(14)	1·479 (5)	C(21)–C(22)	1·377 (5)
C(1)–C(2)	1·393 (6)	C(22)–C(23)	1·372 (5)
C(1)–C(6)	1·380 (5)	C(23)–C(24)	1·368 (6)
C(2)–C(3)	1·392 (6)	C(24)–C(25)	1·361 (6)
C(3)–C(4)	1·360 (6)	C(25)–C(26)	1·378 (6)
C(4)–C(5)	1·394 (5)	C(26)–C(21)	1·379 (5)
C(5)–C(6)	1·386 (5)		

O(1)–S(1)–O(2)	118·6 (2)	N(2)–C(8)–C(9)	107·1 (3)
O(1)–S(1)–N(1)	106·3 (2)	N(3)–C(8)–C(7)	105·8 (3)
O(1)–S(1)–C(21)	107·8 (2)	N(3)–C(8)–C(9)	104·3 (3)
O(2)–S(1)–N(1)	110·0 (2)	C(7)–C(8)–C(9)	115·9 (3)
O(2)–S(1)–C(21)	106·8 (2)	O(4)–C(9)–C(8)	104·4 (3)
N(1)–S(1)–C(21)	106·9 (2)	O(4)–C(9)–C(10)	107·8 (3)
O(3)–S(2)–O(4)	109·4 (2)	O(4)–C(9)–C(13)	105·8 (3)
O(3)–S(2)–N(2)	121·7 (2)	C(8)–C(9)–C(10)	117·6 (3)
O(3)–S(2)–C(15)	106·6 (2)	C(8)–C(9)–C(13)	104·3 (3)
O(4)–S(2)–N(2)	102·8 (2)	C(10)–C(9)–C(13)	115·8 (3)
O(4)–S(2)–C(15)	103·8 (2)	C(9)–C(10)–C(11)	111·9 (3)
N(2)–S(2)–C(15)	111·2 (2)	C(10)–C(11)–C(12)	109·1 (3)
S(2)–O(4)–C(9)	111·0 (2)	C(11)–C(12)–C(7)	112·8 (3)
S(1)–N(1)–C(7)	127·6 (3)	C(9)–C(13)–C(14)	103·2 (3)
S(2)–N(2)–C(8)	112·3 (2)	C(13)–C(14)–N(3)	103·3 (3)
C(5)–N(3)–C(8)	107·4 (3)	S(2)–C(15)–C(16)	121·3 (3)
C(5)–N(3)–C(14)	114·8 (3)	S(2)–C(15)–C(20)	119·3 (3)
C(8)–N(3)–C(14)	107·2 (3)	C(16)–C(15)–C(20)	119·3 (4)
C(6)–C(1)–C(2)	119·0 (3)	C(15)–C(16)–C(17)	120·5 (3)
C(1)–C(2)–C(3)	120·0 (3)	C(16)–C(17)–C(18)	120·8 (4)
C(2)–C(3)–C(4)	120·8 (3)	Cl(2)–C(18)–C(17)	120·8 (4)
C(3)–C(4)–C(5)	119·6 (3)	Cl(2)–C(18)–C(19)	119·9 (4)
C(4)–C(5)–C(6)	120·0 (4)	C(17)–C(18)–C(19)	119·3 (4)
C(4)–C(5)–N(3)	127·6 (3)	C(18)–C(19)–C(20)	120·4 (4)
N(3)–C(5)–C(6)	112·5 (3)	C(19)–C(20)–C(15)	119·6 (4)
C(5)–C(6)–C(1)	120·6 (3)	S(1)–C(21)–C(22)	119·6 (3)
C(5)–C(6)–C(7)	110·0 (3)	S(1)–C(21)–C(26)	120·7 (3)
C(1)–C(6)–C(7)	129·5 (3)	C(22)–C(21)–C(26)	119·8 (4)
N(1)–C(7)–C(6)	112·0 (3)	C(21)–C(22)–C(23)	120·0 (3)
N(1)–C(7)–C(8)	106·4 (3)	C(22)–C(23)–C(24)	119·6 (3)
N(1)–C(7)–C(12)	109·5 (3)	Cl(1)–C(24)–C(23)	119·1 (3)
C(6)–C(7)–C(8)	101·2 (3)	Cl(1)–C(24)–C(25)	119·7 (3)
C(6)–C(7)–C(12)	113·1 (3)	C(23)–C(24)–C(25)	121·2 (4)
C(8)–C(7)–C(12)	114·2 (3)	C(24)–C(25)–C(26)	119·5 (3)
N(2)–C(8)–N(3)	112·3 (3)	C(25)–C(26)–C(21)	119·9 (3)
N(2)–C(8)–C(7)	111·5 (3)		

molecules are shown in Figs. 1, 2, 3 and 4. Details of important molecular planes are given in Table 9 and torsion angles in Table 10.

Table 7. *Compound* (III) ($C_{20}H_{19}ClN_2O_3S$): *bond distances* (Å) *and angles* (°)

Cl(1)–C(18)	1·742 (3)	C(6)–C(7)	1·550 (4)
S(1)–O(1)	1·424 (2)	C(7)–C(13)	1·528 (4)
S(1)–O(2)	1·438 (2)	C(7)–C(14)	1·528 (4)
S(1)–N(1)	1·637 (3)	C(8)–C(9)	1·392 (4)
S(1)–C(15)	1·760 (3)	C(8)–C(13)	1·384 (4)
N(1)–C(7)	1·478 (3)	C(9)–C(10)	1·376 (5)
N(2)–C(1)	1·472 (4)	C(10)–C(11)	1·377 (5)
N(2)–C(3)	1·479 (4)	C(11)–C(12)	1·397 (4)
N(2)–C(12)	1·391 (4)	C(12)–C(13)	1·400 (4)
O(3)–C(14)	1·209 (3)	C(15)–C(16)	1·388 (5)
C(1)–C(2)	1·539 (5)	C(16)–C(17)	1·373 (5)
C(2)–C(3)	1·544 (5)	C(17)–C(18)	1·381 (5)
C(3)–C(4)	1·531 (4)	C(18)–C(19)	1·367 (5)
C(3)–C(14)	1·503 (4)	C(19)–C(20)	1·387 (5)
C(4)–C(5)	1·532 (5)	C(20)–C(15)	1·380 (4)
C(5)–C(6)	1·538 (4)		

O(1)–S(1)–O(2)	120·0 (2)	C(6)–C(7)–C(14)	102·6 (2)
O(1)–S(1)–N(1)	111·4 (1)	C(13)–C(7)–C(14)	112·3 (2)
O(1)–S(1)–C(15)	106·9 (2)	C(13)–C(8)–C(9)	121·2 (3)
O(2)–S(1)–N(1)	105·6 (1)	C(8)–C(9)–C(10)	119·0 (3)
O(2)–S(1)–C(15)	106·8 (1)	C(9)–C(10)–C(11)	121·1 (3)
N(1)–S(1)–C(15)	105·2 (1)	C(10)–C(11)–C(12)	119·9 (3)
S(1)–N(1)–C(7)	121·4 (2)	N(2)–C(12)–C(11)	120·0 (3)
C(1)–N(2)–C(3)	93·2 (2)	N(2)–C(12)–C(13)	120·4 (3)
C(1)–N(2)–C(12)	126·1 (3)	C(11)–C(12)–C(13)	119·5 (3)
C(3)–N(2)–C(12)	122·0 (2)	C(7)–C(13)–C(8)	121·0 (3)
N(2)–C(1)–C(2)	89·2 (3)	C(7)–C(13)–C(12)	120·1 (3)
C(1)–C(2)–C(3)	88·1 (3)	C(8)–C(13)–C(12)	118·9 (3)
N(2)–C(3)–C(2)	88·8 (2)	O(3)–C(14)–C(3)	124·2 (3)
N(2)–C(3)–C(4)	112·3 (3)	O(3)–C(14)–C(7)	123·7 (3)
N(2)–C(3)–C(14)	112·7 (2)	C(3)–C(14)–C(7)	111·2 (2)
C(2)–C(3)–C(4)	119·7 (3)	S(1)–C(15)–C(16)	120·7 (3)
C(2)–C(3)–C(14)	118·6 (3)	S(1)–C(15)–C(20)	119·3 (2)
C(4)–C(3)–C(14)	104·5 (3)	C(16)–C(15)–C(20)	120·0 (3)
C(3)–C(4)–C(5)	110·7 (3)	C(15)–C(16)–C(17)	120·5 (3)
C(4)–C(5)–C(6)	113·2 (3)	C(16)–C(17)–C(18)	118·4 (3)
C(5)–C(6)–C(7)	111·4 (3)	Cl(1)–C(18)–C(17)	119·1 (3)
N(1)–C(7)–C(6)	107·8 (2)	Cl(1)–C(18)–C(19)	118·5 (3)
N(1)–C(7)–C(13)	111·5 (2)	C(17)–C(18)–C(19)	122·3 (3)
N(1)–C(7)–C(14)	111·4 (2)	C(18)–C(19)–C(20)	118·9 (3)
C(6)–C(7)–C(13)	110·8 (2)	C(19)–C(20)–C(15)	119·8 (3)

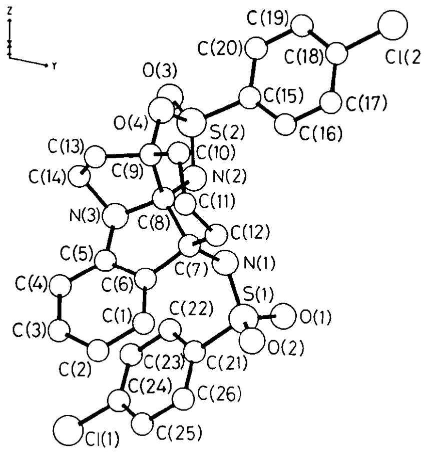

Fig. 2. The molecular structure of compound (II).

Table 8. *Compound* (IV) ($C_{20}H_{20}ClN_3O_4S.C_5H_5N$): *bond distances* (Å) *and angles* (°)

Cl(1)–C(18)	1·744 (5)	C(7)–C(10)	1·505 (5)
S(1)–O(1)	1·433 (3)	C(9)–C(10)	1·378 (5)
S(1)–O(2)	1·434 (3)	C(9)–C(14)	1·386 (5)
S(1)–N(1)	1·621 (3)	C(10)–C(11)	1·372 (5)
S(1)–C(15)	1·765 (4)	C(11)–C(12)	1·390 (6)
O(3)–C(8)	1·215 (4)	C(12)–C(13)	1·377 (7)
O(4)–N(3)	1·369 (5)	C(13)–C(14)	1·388 (6)
N(1)–C(7)	1·461 (5)	C(15)–C(16)	1·388 (6)
N(2)–C(1)	1·457 (5)	C(16)–C(17)	1·375 (6)
N(2)–C(8)	1·372 (5)	C(17)–C(18)	1·374 (6)
N(2)–C(9)	1·414 (5)	C(18)–C(19)	1·364 (6)
N(3)–C(3)	1·297 (6)	C(19)–C(20)	1·378 (6)
C(1)–C(2)	1·529 (6)	C(20)–C(15)	1·390 (6)
C(2)–C(3)	1·531 (7)	N(21)–C(22)	1·312 (7)
C(3)–C(4)	1·497 (7)	N(21)–C(26)	1·328 (7)
C(4)–C(5)	1·510 (7)	C(22)–C(23)	1·326 (8)
C(5)–C(6)	1·536 (6)	C(23)–C(24)	1·311 (9)
C(6)–C(7)	1·564 (5)	C(24)–C(25)	1·331 (9)
C(7)–C(8)	1·532 (6)	C(25)–C(26)	1·393 (8)

O(1)–S(1)–O(2)	120·4 (2)	N(2)–C(8)–C(7)	107·4 (3)
O(1)–S(1)–N(1)	110·3 (2)	N(2)–C(9)–C(10)	110·2 (3)
O(1)–S(1)–C(15)	106·2 (2)	N(2)–C(9)–C(14)	127·5 (4)
O(2)–S(1)–N(1)	105·5 (2)	C(10)–C(9)–C(14)	122·3 (4)
O(2)–S(1)–C(15)	107·7 (2)	C(7)–C(10)–C(9)	108·2 (3)
N(1)–S(1)–C(15)	105·9 (2)	C(7)–C(10)–C(11)	131·3 (4)
S(1)–N(1)–C(7)	120·9 (3)	C(9)–C(10)–C(11)	120·2 (4)
C(1)–N(2)–C(8)	121·6 (3)	C(10)–C(11)–C(12)	118·7 (4)
C(1)–N(2)–C(9)	124·7 (3)	C(11)–C(12)–C(13)	120·6 (4)
C(8)–N(2)–C(9)	110·3 (3)	C(12)–C(13)–C(14)	121·4 (4)
O(4)–N(3)–C(3)	108·8 (4)	C(13)–C(14)–C(9)	116·9 (4)
N(2)–C(1)–C(2)	109·8 (4)	S(1)–C(15)–C(16)	120·5 (3)
C(1)–C(2)–C(3)	112·9 (4)	S(1)–C(15)–C(20)	120·0 (3)
N(3)–C(3)–C(2)	123·0 (5)	C(16)–C(15)–C(20)	119·5 (4)
N(3)–C(3)–C(4)	113·2 (4)	C(15)–C(16)–C(17)	120·4 (4)
C(2)–C(3)–C(4)	123·7 (4)	C(16)–C(17)–C(18)	118·8 (4)
C(3)–C(4)–C(5)	119·9 (4)	Cl(1)–C(18)–C(17)	118·9 (4)
C(4)–C(5)–C(6)	119·3 (4)	Cl(1)–C(18)–C(19)	119·1 (4)
C(5)–C(6)–C(7)	116·1 (3)	C(17)–C(18)–C(19)	121·9 (4)
N(1)–C(7)–C(6)	107·9 (3)	C(18)–C(19)–C(20)	119·4 (4)
N(1)–C(7)–C(8)	114·7 (3)	C(19)–C(20)–C(15)	119·9 (4)
N(1)–C(7)–C(10)	115·8 (3)	C(22)–N(21)–C(26)	115·8 (5)
C(6)–C(7)–C(8)	105·7 (3)	N(21)–C(22)–C(23)	124·6 (6)
C(6)–C(7)–C(10)	109·8 (3)	C(22)–C(23)–C(24)	119·4 (6)
C(8)–C(7)–C(10)	102·5 (3)	C(23)–C(24)–C(25)	120·5 (6)
O(3)–C(8)–N(2)	126·0 (4)	C(24)–C(25)–C(26)	117·6 (5)
O(3)–C(8)–C(7)	126·4 (4)	C(25)–C(26)–N(21)	122·1 (5)

Table 9. *Equations of least-squares best planes and, in square brackets, distances* (Å) *of atoms from the planes*

The equations are referred to the crystal axes and x, y and z are fractional coordinates. E.s.d.'s of distances <0·005 Å.

Compound (I)

Plane (1): Cl(1), C(15) to C(20)

$$7{\cdot}730x + 5{\cdot}824y + 0{\cdot}295z = 8{\cdot}604$$

[Cl(1) –0·005, C(15) –0·003, C(16) –0·001, C(17) 0·005, C(18) 0·001, C(19) 0·007, C(20) –0·003, S(1)* –0·019, O(1)* 0·828, O(2)* 0·254]

Plane (2): N(2), C(7) to C(14)

$$7{\cdot}487x - 2{\cdot}082y + 4{\cdot}949z = 6{\cdot}459$$

[N(2) 0·027, C(7) –0·042, C(8) –0·025, C(9) 0·032, C(10) 0·026, C(11) –0·016, C(12) –0·032, C(13) –0·006, C(14) 0·036]

Compound (II)

Plane (3): Cl(2), C(15) to C(20)

$$7{\cdot}457x - 5{\cdot}791y + 1{\cdot}917z = 1{\cdot}561$$

[Cl(2) 0·016, C(15) 0·022, C(16) 0·001, C(17) –0·020, C(18) 0·001, C(19) –0·014, C(20) –0·006, S(2)* 0·166, N(2)* –0·209, O(3)* –0·437, O(4)* 1·731]

Plane (4): Cl(1), C(21) to C(26)

$$-5{\cdot}724x + 9{\cdot}116y - 4{\cdot}326z = 3{\cdot}059$$

[Cl(1) –0·035, C(21) –0·031, C(22) 0·001, C(23) 0·023, C(24) 0·020, C(25) 0·028, C(26) –0·005, S(1)* –0·103, O(1)* 1·164, O(2)* –0·569]

Plane (5): C(1) to C(6)

$$-7{\cdot}025x + 5{\cdot}535y - 4{\cdot}986z = -3{\cdot}870$$

[C(1) –0·013, C(2) –0·002, C(3) 0·015, C(4) –0·012, C(5) –0·003, C(6) 0·016, C(7)* 0·059, N(3)* 0·021, C(8)* –0·222]

Plane (6): C(9), O(4), S(2), N(2)

$$-3{\cdot}998x - 10{\cdot}046y - 0{\cdot}192z = -10{\cdot}688$$

[C(9) –0·011, O(4) 0·018, S(2) –0·017, N(2) 0·011, C(8)* 0·239]

Plane (7): N(3), C(8), C(9), C(13)

$$-5{\cdot}402x - 1{\cdot}419y + 9{\cdot}618z = 0{\cdot}502$$

[N(3) 0·030, C(8) –0·045, C(9) 0·044, C(13) –0·030, C(14)* 0·600]

Compound (III)

Plane (8): Cl(1), C(15) to C(20)

$$2{\cdot}959x + 8{\cdot}232y - 2{\cdot}531z = 2{\cdot}322$$

[Cl(1) –0·029, C(15) –0·025, C(16) –0·002, C(17) 0·023, C(18) 0·013, C(19) 0·025, C(20) –0·004, S(1)* –0·078, O(1)* –0·423, O(2)* 1·152]

Plane (9): C(8) to C(13)

$$-4{\cdot}169x - 5{\cdot}403y + 9{\cdot}284z = -1{\cdot}193$$

[C(8) 0·014, C(9) 0·018, C(10) –0·025, C(11) –0·001, C(12) 0·032, C(13) –0·038]

Plane (10): C(1), C(2), C(3), N(2)

$$-1{\cdot}715x - 8{\cdot}666y + 6{\cdot}771z = 1{\cdot}556$$

[C(1) –0·042, C(2) 0·040, C(3) –0·042, N(2) 0·044]

Compound (IV)

Plane (11): Cl(1), C(15) to C(20)

$$-6{\cdot}835x - 1{\cdot}630y + 6{\cdot}227z = 2{\cdot}128$$

[Cl(1) –0·016, C(15) –0·016, C(16) –0·010, C(17) 0·024, C(18) 0·005, C(19) 0·005, C(20) 0·009, S(1)* –0·066, O(1)* 0·128, O(2)* 0·823]

(*a*) *Compound* (I)

The crystal contains isolated molecules which have configuration and dimensions consistent with formula (I). There are no unduly short contacts between molecules, nor any hydrogen bonds formed at the amino N(1) atom.

The dimensions of the *p*-chlorophenylsulphonylamino residue agree well with the corresponding values in 1-methyl-3-(*p*-tolylsulphonylamino)indole (Cameron, Prout, Denton, Spagna & White, 1975) where the sulphonylamino group is conjugated through N(1) both to a phenyl group and to N(2). It is suggested that, in both compounds, the d_π–p_π inter-

Table 9 (*cont.*)

Plane (12): C(9) to C(14)

$$4{\cdot}285x - 0{\cdot}747y - 9{\cdot}464z = -4{\cdot}441$$

[C(9) −0·004, C(10) −0·002, C(11) 0·007, C(12) −0·006, C(13) −0·000, C(14) 0·005, C(7)* 0·129, C(8)* −0·064, N(2)* −0·009]

Plane (13): N(21) to C(26); pyridine solvate

$$7{\cdot}432x + 10{\cdot}807y + 5{\cdot}292z = 3{\cdot}746$$

[N(21) −0·002, C(22) 0·006, C(23) −0·004, C(24) −0·002, C(25) 0·006, C(26) −0·004]

* Atoms not included in the plane calculation.

action between the S and N atoms is the dominant effect, rather than electron delocalization from this N atom into the indole fragment. The dihedral angles about N(1)–S(1) and the S(1)–O distances are similar to those found in 1-methyl-3-(*p*-tolylsulphonylamino)-indole, and reflect the attraction between S–O and N–H dipoles of opposite polarity.

The indole nucleus is essentially coplanar [plane (2) in Table 9]; maximum deviations from the mean plane are at C(14) +0·036 and C(12) −0·032 Å. The bond lengths and angles are much as expected; the steric strain of bonding a five- to a six-membered ring causes the bond between C(9) and C(14) to increase somewhat from the usual aromatic C–C length, and the lengths C(9)–C(8) (1·431 Å) and C(8)–C(7) (1·371 Å) indicate that some of the formal double-bond character is retained by C(8)–C(7).

As is illustrated by the torsion angles, Table 10, the N(2)–C(7) azocine ring is non-planar and is folded so as to envelope the O atom at C(3). With Hendrickson's (1964) notation, the conformation of the ring can best be described as *BC*. In the ring, while the C–C lengths and the N(2)–C(1) distance agree with the expected single-bond values, the N(2)–C(7) length of 1·377 Å indicates some partial double-bond character. Also, the heterocyclic ring bond angles, with the exception of C(2)–C(3)–C(4) which involves an atom outside the ring, are all greater than expected, illustrating that the ring is strained. This effect has been found previously (Clearfield, Jones, Kellum & Saldarriaga-Molina, 1975).

(*b*) *Compound* (II)

The crystal contains isolated molecules which have configuration and dimensions consistent with formula

Table 10. *Torsion angles* (°)

The torsion angle, *I–J–K–L*, about the bond *J–K* is defined as the angle the bond *K–L* is rotated from the *IJK* plane. It is positive when, on looking from *IJ* to *KL*, the rotation is clockwise. Torsion angle e.s.d.'s are <0·6° (Shmueli, 1974, eq. 8).

Compound (I)					
C(8)–N(1)–S(1)–O(1)	173	N(1)–C(8)–C(7)–C(6)	4	C(2)–C(3)–C(4)–C(5)	−110
C(8)–N(1)–S(1)–O(2)	44	C(5)–C(4)–C(3)–O(3)	70	C(3)–C(4)–C(5)–C(6)	73
S(1)–N(1)–C(8)–C(7)	−99	C(1)–C(2)–C(3)–O(3)	−125	C(4)–C(5)–C(6)–C(7)	−71
S(1)–N(1)–C(8)–C(9)	88	C(6)–C(7)–N(2)–C(14)	−176	C(5)–C(6)–C(7)–N(2)	79
H(1)–N(1)–C(8)–C(9)	−55	N(2)–C(1)–C(2)–C(3)	54	C(6)–C(7)–N(2)–C(1)	−5
N(1)–C(8)–C(7)–N(2)	−175	C(1)–C(2)–C(3)–C(4)	55	C(7)–N(2)–C(1)–C(2)	−91
Compound (II)					
C(7)–N(1)–S(1)–O(1)	−170	N(2)–C(8)–C(9)–O(4)	16	C(7)–C(8)–C(9)–C(10)	22
C(7)–N(1)–S(1)–O(2)	−41	N(2)–C(8)–C(9)–C(10)	−103	C(8)–C(9)–C(10)–C(11)	−40
S(1)–N(1)–C(7)–C(6)	−42	C(8)–C(9)–O(4)–S(2)	−11	C(9)–C(10)–C(11)–C(12)	61
S(1)–N(1)–C(7)–C(8)	−152	C(9)–O(4)–S(2)–N(2)	3	C(10)–C(11)–C(12)–C(7)	−65
S(1)–N(1)–C(7)–C(12)	84	C(9)–O(4)–S(2)–C(15)	−113	C(11)–C(12)–C(7)–C(8)	46
S(2)–N(2)–C(8)–C(9)	−15	O(4)–S(2)–N(2)–C(8)	8	C(12)–C(7)–C(8)–C(9)	−24
Compound (III)					
C(7)–N(1)–S(1)–O(1)	−12	C(7)–C(14)–C(3)–N(2)	50	C(2)–C(3)–N(2)–C(12)	−144
C(7)–N(1)–S(1)–O(2)	−144	C(7)–C(14)–C(3)–C(2)	152	C(14)–C(3)–C(4)–C(5)	58
S(1)–N(1)–C(7)–C(14)	64	C(14)–C(3)–N(2)–C(12)	−23	C(3)–C(4)–C(5)–C(6)	−51
S(1)–N(1)–C(7)–C(13)	−63	C(14)–C(3)–N(2)–C(1)	114	C(4)–C(5)–C(6)–C(7)	52
N(1)–C(7)–C(14)–O(3)	17	C(4)–C(3)–N(2)–C(1)	−128	C(5)–C(6)–C(7)–C(14)	−58
N(1)–C(7)–C(14)–C(3)	−174	C(4)–C(3)–N(2)–C(12)	95	C(6)–C(7)–C(14)–C(3)	71
C(7)–C(14)–C(3)–C(4)	−72	C(2)–C(3)–N(2)–C(1)	−7		
Compound (IV)					
C(7)–N(1)–S(1)–O(1)	29	C(5)–C(4)–C(3)–N(3)	−136	C(4)–C(5)–C(6)–C(7)	−98
C(7)–N(1)–S(1)–O(2)	160	C(1)–N(2)–C(8)–O(3)	−24	C(5)–C(6)–C(7)–C(8)	61
S(1)–N(1)–C(7)–C(6)	−176	C(6)–C(7)–C(8)–O(3)	72	C(6)–C(7)–C(8)–N(2)	−104
S(1)–N(1)–C(7)–C(8)	−59	C(1)–C(2)–C(3)–C(4)	−109	C(7)–C(8)–N(2)–C(1)	151
S(1)–N(1)–C(7)–C(10)	60	C(2)–C(3)–C(4)–C(5)	48	C(8)–N(2)–C(1)–C(2)	−81
C(1)–C(2)–C(3)–N(3)	76	C(3)–C(4)–C(5)–C(6)	80	N(2)–C(1)–C(2)–C(3)	61

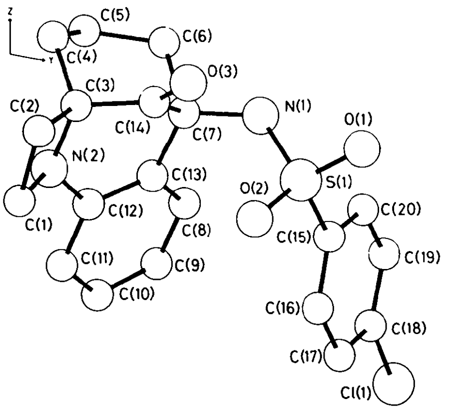

Fig. 3. The molecular structure of compound (III) seen in projection down *a*.

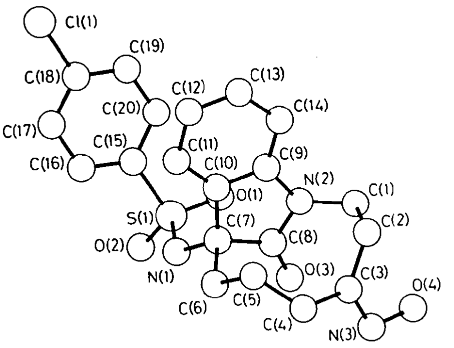

Fig. 4. The molecular structure of compound (IV).

(II). There are no unduly short contacts between molecules, nor any hydrogen bonds formed at the amino N(1).

The major point of interest in the structure is the appearance of the oxathiazoline ring, C(8)–C(9)–O(4)–S(2)–N(2). Recently (L'Abbé, Yu, Declercq, Germain & Van Meerssche, 1978) the crystal structure of 4-*p*-tolylimino-2,5,5-trimethyl-4,5-dihydro-1,$2\lambda^6$,3-oxathiazole 2-oxide has been determined, and the mechanism for its formation is analogous to that proposed for (II) (Bailey, Scott & Vandrevala, 1980). Although this mode of decomposition is known for acyl azides (Graziano & Scarpati, 1976) it is without precedent for sulphonyl azides (L'Abbé, 1969). In the oxathiazole 2-oxide the oxathiazoline ring is planar, while in (II) it exhibits an envelope-type conformation with four atoms coplanar and C(8) 0·239 Å out of plane [plane (6), Table 9]. In general, the distances and angles within the rings are in close agreement in the two structures, except that N(2)–C(8) and C(8)–C(9) are longer in (II) than the corresponding distances in the oxathiazole 2-oxide presumably because of electron delocalization from the tolylimino group into the ring in the latter, similar to that found previously (Cameron, Prout, Denton, Spagna & White, 1975).

Both chlorobenzene rings are planar [planes (3) and (4) in Table 9]. In the *p*-chlorophenylsulphonylamino residue S(1)–N(1) is shorter (1·599 Å) and N(1)–C(7) longer (1·498 Å) than the corresponding distances in (I). This effect is apparently due to increased p_π–d_π bonding (Bailey, Baldrey, Peach, Critchley, Prout & White, 1976), and the angle between the C(7)–N(1)–S(1) and O(1)–S(1)–O(2) planes (53·5°) is suitable for such an interaction, as is the angle at N(1) (127·6°). The dihedral angles about N(1)–S(1) (Table 10) are similar to those found in (I) and other related sulphonylamino systems (Critchley, Prout, Watkin, Bailey & Peach, 1977).

The benzene ring C(1) to C(6) is planar [plane (5) in Table 9] (maximum deviation +0·016 Å) and both N(3) and C(7) lie close to this plane (+0·021 and +0·059 Å respectively) but C(8) is further out of it (−0·222 Å). This non-planarity is due to the distortion necessary to fuse the five rings. The N(3)–C(5) length (1·404 Å) shows that this bond retains some formal double-bond character, while the C(7)–C(8) and C(8)–C(9) lengths (1·578 and 1·593 Å, respectively) illustrate the lengthening observed in crowded molecules containing tetra-substituted carbon (Oh & Maslen, 1966).

The five-membered ring N(3), C(14), C(13), C(8), C(9) has an envelope-type conformation with four atoms approximately coplanar and C(14) 0·600 Å out of plane [plane (7) in Table 9]. The ring C(7) to C(12) has the chair conformation, and C–C lengths [except those involving C(8)] and interbond angles have the values expected for a cyclohexane-like system.

(*c*) *Compound* (III)

The crystals are built up from isolated molecules of formula (III) which neither make unduly short contacts with their neighbours nor form hydrogen bonds at the amino N(1).

Fig. 3 demonstrates that the saturated ring C(3)–C(4)–C(5)–C(6)–C(7)–C(14) adopts the chair conformation, and the C–C lengths and interbond angles have values as expected for a cyclohexane-like system. The dimensions and conformation of the two fused six-membered rings C(8) to C(13), N(2), C(3), C(14), C(7) are in close agreement with those found in *cis*-5,6,6a,7,8,9,10,10a-octahydro-5-methyl-10a-(*p*-tolylsulphonylamino)-6-(*p*-tolylsulphonylimino)phenanthridine (Critchley, Prout, Watkin, Bailey & Peach, 1977).

The dimensions of the *p*-chlorophenylsulphonylamino residue at C(7) agree with corresponding values in the related compound 1,2,3,4-tetrahydro-1,2,4-trimethyl-4-(*p*-tolylsulphonylamino)-3-(*p*-tolylsulphonylimino)quinoline (Cameron, Prout, Denton, Spagna & White, 1975) where there is also no opportunity for π-electron delocalization from S through N to an aromatic system.

The azetidine ring N(2)—C(1)—C(2)—C(3) exhibits a slightly buckled conformation [plane (10) in Table 9], similar to that found in L-azetidine-2-carboxylic acid (Berman, McGandy, Burgner & VanEtten, 1969).

(*d*) *Compound* (IV)

The crystal contains isolated molecules which have configuration and dimensions consistent with formula (IV). There are no unduly short contacts between molecules, nor any hydrogen bonds formed at the amino N(1). The pyridine solvent molecule is planar [plane (13) in Table 9] and interatomic distances and angles are normal but with somewhat high e.s.d.'s probably a consequence of high thermal motion.

The dimensions of the *p*-chlorophenylsulphonylamino residue at C(7) agree with corresponding values in (III). In both these compounds, there is no opportunity for π-electron delocalization from S through N to an aromatic system.

While the benzene ring C(9)–C(14) is planar [plane (12) in Table 9] (maximum deviation +0·007 Å) and N(2) lies close to this plane (−0·009 Å), both C(7) and C(8) are further out of it (+0·129 and −0·064 Å, respectively). This non-planarity, like that found in (II), is due to the distortion necessary to fuse the three rings. In general, the dimensions of the indoline group agree well with those found in 1-methyl-2-picrylimino-indoline (Bailey, Warr, Allison & Prout, 1970).

In the ring N(2), C(1) to C(8) bond lengths and angles are much as expected; the oxime group is not unusual; the C(6)—C(7) length, 1·564 (5) Å, illustrates the lengthening observed in crowded molecules containing tetra-substituted carbon. With Hendrickson's (1964) notation, the conformation of the nine-membered ring can best be described as twist-chair–chair (TCC) (see Table 10).

We thank the SRC for support for CC.

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