Fig. 2. The ring torsion angles and asymmetry parameters (°) (Duax & Norton, 1975). $\Delta C_s^P = 2\cdot3$ (8)°, $\Delta C_2^{C(4)} = 7\cdot7$ (8)°.

The C(7)—N and C(6)—C(4) bonds form angles of 102·9 (9) and 39·4 (9)° with the normal to the least-squares plane [N(1),O,C(4),C(5)].

The oxazaphospholidine ring is almost an ideal envelope with the P atom deviating by −0·339 (2) Å from the least-squares plane [N(1),O,C(4),C(5)], Fig. 1. The angle between this plane and the [O,P,N(1)] plane is 18·2 (9)°. The substituents at the aniline N atom are nearly coplanar ($\sum = 358°$). The plane [P,N(2),C(20)] is almost exactly perpendicular to the [N(1),O,C(5),C(4)] plane; the angle between normals to these planes is 88·1 (9)°. The S—P bond is practically coplanar with the plane [P,N(2),C(20)] and forms the angle of 89·8 (6)° with the normal to this plane. The plane is also very nearly coplanar with a pseudo plane of symmetry passing through C(23),C(20) and the middle of the C(4)—C(5) bond. It is illustrated by the distances

of the following atoms from the plane: −1·147 (7) and 1·098 (10) Å for C(22) and C(24), −1·217 (5) and 1·166 (6) Å for O and N(1), −0·844 (8) and 0·707 (9) Å for C(5) and C(4). The phenyl rings show normal geometry. The normals to them form the angles 108 (1) and 102 (1)° respectively with the normal to the [N(1),O,C(5),C(4)] plane.

Fig. 2 shows torsion angles and asymmetry parameters for the oxazaphospholidine ring.

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Structure of p-(p-Nitroanilino)phenyl Isothiocyanate, C₁₃H₉N₃O₂S*

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Abstract. $M_r = 271\cdot30$, monoclinic, $P2_1/n$, $a = 9\cdot1998$ (3), $b = 10\cdot6690$ (4), $c = 12\cdot6690$ (4) Å, $\beta = 98\cdot725$ (2)°, $V = 1229\cdot1$ Å³, $Z = 4$, $D_m = 1\cdot44$ (2), $D_x = 1\cdot466$ g cm⁻³, $\lambda(\text{Cu } K\alpha_1) = 1\cdot54056$ Å, $\mu = 23\cdot0$ cm⁻¹, $F(000) = 560$, $T = 296$ (1) K. Final $R(F) = 0\cdot039$ for 1964 counter reflections. The two planar benzene rings, connected by an imino N atom, make an angle of 47·28 (6)° with each other. The SCNC group is nearly linear, with S—C—N and C—N—C angles of 178·1 (2) and 168·9 (3)° respectively. The C—NH—C group is nearly planar; the C—N—C angle has the large value 127·6 (1)°, due mainly to steric repulsion between the two aromatic rings.

Introduction. Hycanthone is a drug still widely used in Africa, the Middle East, and Brazil for the treatment of schistosomiasis despite its potential risks to human health implied by the carcinogenic, mutagenic, and teratogenic activities observed in experimental animals

and bacteria (Bueding & Batzinger, 1977). In a continued search for safer drugs, Bueding (1975) reported that alterations of the hycanthone molecule to benzothiopyranoindazole derivatives yielded marked reduction in mutagenic activity without decreasing antischistosomal potency. Following the X-ray structural study of hycanthone methanesulfonate (Wei & Einstein, 1978), crystal structures of three such hycanthone analogs, including the methanesulfonate salt of the chlorobenzothiopyranoindazole derivative [designated IA-4 (Bueding, Fischer & Bruce, 1973)] (Wei, 1981), the dechloro form of IA-4 (Wei, 1982), and the 1:1 chloroform solvate of IA-4 (Wei, 1983) have been subsequently determined in this laboratory. The results revealed that the thioxanthene ring system is invariably roughly planar and that the terminal N of the side chain is responsible for the formation of various inter-molecular hydrogen bonds, thus supporting the hypotheses made for this family of antischistosomal drugs regarding drug–DNA interaction (for review, see Weinstein & Hirschberg, 1971; Hirschberg, 1975).

Recently, a new type of antischistosomicidal drug, p-(p-nitroanilino)phenyl isothiocyanate (amoscanate), was found to exhibit a high chemotherapeutic index without appreciable toxicity (Striebel, 1976, 1978). This discovery has prompted many biological studies around the world. The therapeutic use of amoscanate, however, is hampered because mutagenic materials are produced from it by intestinal microorganisms (Molineaux, Batzinger, Schmidt & Bueding, 1980). This mutagenic activity may be prevented by coadministration of erythromycin, an enteric antibacterial agent (Batzinger, Bueding, Crawford & Bruce, 1979).

Although X-ray structures of many metal complexes containing the isothiocyanate group as a ligand have been reported, no three-dimensional X-ray study of an organic molecule containing the group has appeared in the literature, to the best of our knowledge. We report here the structural investigation of amoscanate, in which the isothiocyanate group is attached to an aromatic ring system.

Experimental. Sample of amoscanate powder (product of Ciba–Geigy Ltd) kindly supplied by Professor Ernest Bueding; clear-yellow diamond-shaped crystals grown from a solution in a 1:1 mixture of ethyl acetate and chloroform, crystal density determined by flotation in mixtures of chloroform and 1,2,3-trichloropropane, crystal with approximate dimensions $0.2 \times 0.2 \times 0.1$ mm mounted on a Picker four-circle diffractometer; angle measurements of 12 strong reflections in 2θ range 110–127° used in least-squares refinement of lattice parameters (Busing, Ellison, Levy, King & Roseberry, 1968); systematic absences $h0l$, $h + l = 2n + 1$, $0k0$, $k = 2n + 1$ consistent with $P2_1/n$; intensity data collected by θ–2θ scans in 2θ range 2–130° divided into five separate subranges, within any subrange maximum

variation for standard reflections $\pm 2\%$; of 2095 unique nonzero reflections, 129 with $F_o^2 < \sigma(F_o^2)$ and two with $|\Delta F^2| > 15\sigma(F_o^2)$ excluded from final least-squares refinement; absorption corrections ranging from 0.86 to 1.00 applied empirically from a transmission curve obtained by measuring intensities of the 101 reflection for rotation angles at 10° intervals about its reciprocal lattice vector, maximum mosaic spread estimated to be 0.35°.

Attempts to locate S position from a Patterson map were unsuccessful; structure solved with *MULTAN* (Germain, Main & Woolfson, 1971) using seven reflections in starting set; preliminary refinements carried out using block-diagonal least-squares program of Shiono (1971), positions of the nine H atoms obtained as those of the nine strongest peaks in a subsequent difference Fourier map; final four cycles of least-squares refinement on F^2 carried out with full-matrix program *ORXFLS4* (Busing, Martin & Levy, 1962); scattering factors taken from those of Cromer & Waber (1974), anomalous-scattering corrections for S (Cromer, 1974) applied; weights (w) used were reciprocals of variances $\sigma^2(F_o^2)$, estimated according to empirical expressions given elsewhere (Wei & Einstein, 1978); H atoms treated isotropically and non-H atoms anisotropically; on final cycle largest parameter shift 0.04σ, average shift 0.003σ; for the 1964 reflections utilized in refinement, $wR(F^2)$ [$= (\sum w|\Delta F^2|^2 / \sum wF_o^4)^{1/2}$] $= 0.113$ and $S = 2.24$ { S defined as $[\sum w|\Delta F^2|^2/(n - p)]^{1/2}$ where n is the number of observations and p is the number of variables}; with all 2095 unique nonzero reflections included $R(F) = 0.041$; a final difference Fourier synthesis exhibited no residual peaks greater than 0.15 e Å^{-3}.

Discussion. Positional parameters and isotropic temperature factors (or their equivalents) are presented in Table 1.*

The crystal structure consists of individual molecules of the configuration depicted in Fig. 1 [prepared with the program *ORTEP* II (Johnson, 1976)]. Bond lengths, bond angles, and their corresponding e.s.d.'s were calculated by the program *ORFFE4* (Busing, Martin & Levy, 1964), and are given in the figure.

The $-C_6H_4NO_2$ and $-C_6H_4NCS$ fragments are connected by $N(2)$, the $C(5)-N(2)-C(8)$ angle being 127.6 (1)°. Each benzene ring is essentially planar: the C atoms in the two benzene rings deviate from the least-squares planes by less than 0.009 (1) Å, while substituent atoms on the rings deviate as much as 0.074 (3) Å. In order to minimize intramolecular

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* Lists of structure factors, anisotropic thermal parameters and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38326 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional and isotropic thermal parameters*

The e.s.d.'s for the least significant figures are given in parentheses in all tables and in the text. The equivalent isotropic temperature factors for C, N, O and S atoms were calculated by the relation $B_{eq} = \frac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + 2\beta_{13}ac\cos\beta)$ (Hamilton, 1959).

|	x	y	z	B_{eq} or B (Å²)
S	0·51059 (7)	0·48561 (6)	0·85541 (5)	6·85 (2)
C(1)	0·4204 (2)	0·4716 (2)	0·7404 (2)	4·90 (4)
C(2)	0·2605 (2)	0·4404 (2)	0·5610 (1)	5·17 (5)
C(3)	0·1368 (2)	0·3679 (2)	0·5630 (1)	5·41 (5)
C(4)	0·0446 (2)	0·3429 (2)	0·4689 (1)	4·89 (4)
C(5)	0·0741 (2)	0·3925 (1)	0·3726 (1)	4·22 (3)
C(6)	0·1976 (2)	0·4669 (2)	0·3720 (1)	4·40 (4)
C(7)	0·2911 (2)	0·4900 (2)	0·4661 (2)	4·86 (4)
C(8)	0·0000 (2)	0·3566 (1)	0·1772 (1)	3·93 (3)
C(9)	−0·1178 (2)	0·3650 (1)	0·0932 (1)	4·27 (4)
C(10)	−0·0977 (2)	0·3458 (2)	−0·0105 (1)	4·27 (3)
C(11)	0·0424 (2)	0·3193 (1)	−0·0320 (1)	3·85 (3)
C(12)	0·1603 (2)	0·3121 (2)	0·0494 (1)	4·43 (4)
C(13)	0·1403 (2)	0·3301 (2)	0·1537 (1)	4·40 (4)
N(1)	0·3560 (2)	0·4644 (2)	0·6550 (1)	6·98 (5)
N(2)	−0·0280 (2)	0·3713 (2)	0·2807 (1)	4·74 (4)
N(3)	0·0650 (2)	0·2968 (1)	−0·1410 (1)	4·54 (3)
O(1)	−0·0405 (2)	0·3045 (2)	−0·2128 (1)	6·56 (4)
O(2)	0·1894 (1)	0·2706 (2)	−0·1584 (1)	6·31 (4)
H(3)	0·111 (3)	0·336 (2)	0·626 (2)	6·5 (5)
H(4)	−0·039 (2)	0·295 (2)	0·465 (2)	5·4 (4)
H(6)	0·219 (2)	0·504 (2)	0·310 (2)	5·0 (4)
H(7)	0·373 (2)	0·539 (2)	0·463 (2)	5·2 (4)
H(N2)	−0·115 (3)	0·357 (2)	0·289 (2)	5·4 (4)
H(9)	−0·210 (2)	0·384 (2)	0·110 (2)	5·2 (4)
H(10)	−0·182 (2)	0·352 (2)	−0·067 (2)	5·2 (4)
H(12)	0·256 (3)	0·294 (2)	0·033 (2)	6·5 (5)
H(13)	0·221 (1)	0·325 (2)	0·209 (2)	5·8 (5)
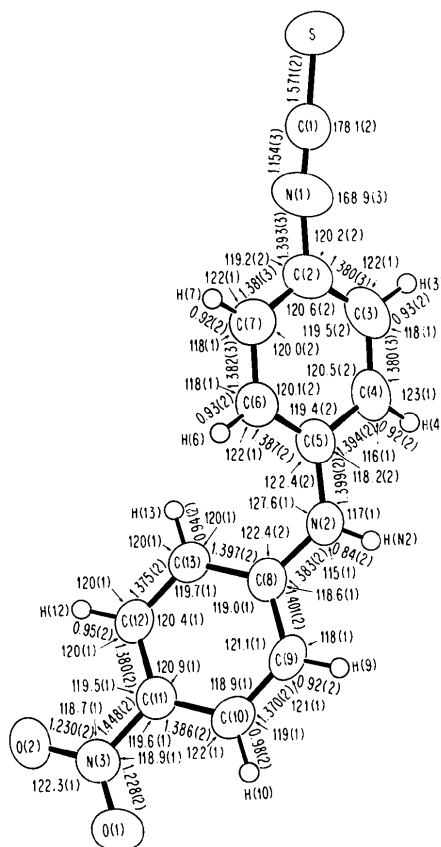
Fig. 1. Bond lengths (Å) and bond angles (°) with their e.s.d.'s for amoscanate.

nonbonded repulsions, the $-C_6H_4NCS$ ring is twisted counterclockwise about the C(5)—N(2) bond (Fig. 1), so that torsion angles C(6)—C(5)—N(2)—C(8) and C(4)—C(5)—N(2)—C(8) are 36·8 (2) and −147·2 (2)° respectively. This gives rise to the angle of 47·28 (6)° between normals to the least-squares planes for the two aromatic rings. A calculation of the least-squares plane for the C—NH—C group shows that the maximum deviation from the plane is that of N(2), 0·033 (7) Å. This suggests a planar-trigonal configuration at N(2), although the precision of location of H(N2) is low.

The C—C distances and C—C—C angles in the phenyl ring adjacent to the SCN group are within three e.s.d.'s of the average values of 1·384 (1) Å and 120·0 (1)°. The distribution of bond distances in the other aromatic ring, however, shows significant variations: the C(9)—C(10) and C(12)—C(13) distances are much shorter than the average of the other four C—C distances, 1·391 (1) Å. This may be due to the 'quinoidal' resonance effect (Domenicano, Vaciago & Coulson, 1975), which occurs when an electron-donating group and an electron-withdrawing group are located *para* to each other. Similar effects have been observed in 4-nitroaniline (Trueblood, Goldish & Donohue, 1961), two crystalline forms of *p*-nitrophenol (Coppens & Schmidt, 1965*a,b*) and *N,N*-dimethyl-*p*-nitroaniline (Mak & Trotter, 1965). The N(2)—C(5) and N(2)—C(8) distances of 1·399 (2) and 1·383 (2) Å

are compatible with the estimated σ-bond length of 1·41 (2) Å between a trigonal nitrogen and a trigonal carbon (Trueblood *et al.*, 1961). However, these distances are significantly shorter than the $C-NO_2$ distance of 1·448 (2) Å (see Vincent & Wheatley, 1972).

The C(2)—N(1)—C(1)—S group in this structure is almost linear. The bond lengths and angles are in close agreement with those reported for many metal complexes containing isothiocyanate groups as ligands (see below) and can be considered as evidence in favor of the $C-N^+\equiv C-S^-$ resonance formulation (Pauling, 1960).

Table 2 shows bond lengths and angles for some selected S—C—N—X groups (X denotes atoms to which N is bound) in a selection of structures. A number of SCN—metal complexes cited in Table 2 are analogous to the present case (X = aromatic C atom) in that there is a π-electron interaction between the N and X atoms, with N as the π-acceptor. The S—C—N angle in each case is larger than the C—N—X angle. While the S—C—N angles fall within 4° (except for HNCS) of 180°, the C—N—X angles show considerably large variations from 180°. In addition to resonance (Paul-

Table 2. *Comparison of bond distances (Å) and angles (°) for selected X*NCS *groups*

Compound	X	Metal coordination	S–C	C–N	S–C–N	C–N–X	Reference
O₂NC₆H₄NHC₆H₄NCS	O₂NC₆H₄NHC₆H₄	—	1·571 (2)	1·154 (3)	178·1 (2)	168·9 (3)	this work
HNCS	H	—	1·5665 (6)	1·207 (2)	174 (2)	132 (2)	(a)
B₁₀H₁₃NCS	B₁₀H₁₃	—	1·581 (4)	1·149 (5)	178·1 (6)	171·0 (6)	(b)
Na₂[Co(NCS)₄].8H₂O*	Co²⁺	Tetrahedral	1·621 (8)	1·152 (10)	178·0 (7)	170·3 (6)	(c)
Ni(C₃NOH₉)₂(NCS)₂(racemic isomer)	Ni²⁺	Octahedral	1·631 (2)	1·149 (3)	177·9 (2)	162·0 (2)	(d)
Co₂(C₈H₇N₃)₅(NCS)₄.2·7H₂O*	Co²⁺	Octahedral	1·63 (1)	1·18 (1)	177·0 (7)	154·5 (5)	(e)
Co(C₂H₃N₃)₂(NCS)₂	Co²⁺	Octahedral	1·635 (6)	1·160 (5)	176 (2)	153·7 (8)	(f)
Cu(C₂H₃N₃)₂(NCS)₂	Cu²⁺	Octahedral	1·634 (3)	1·152 (4)	179·9 (7)	160·9 (3)	(f)
Zn(C₂H₃N₃)₂(NCS)₂	Zn²⁺	Octahedral	1·634 (3)	1·150 (4)	179·0 (2)	153·9 (1)	(f)
Mn₂(C₃H₅N₃)₅(NCS)₄*	Mn²⁺	Octahedral	1·624 (2)	1·147 (3)	178·4 (2)	154·1 (2)	(g)
Na₃[V(NCS)₆].12H₂O	V³⁺	Octahedral	1·631 (3)	1·152 (4)	178·3 (3)	176·4 (2)	(h)
Nb(C₁₀H₈N₂)₂(NCS)₄	Nb⁴⁺	Eight-coordinated	1·599 (3)	1·159 (4)	178·8 (3)	166·5 (3)	(i)
Zr(C₁₀H₈N₂)₂(NCS)₄	Zr⁴⁺	Eight-coordinated	1·590 (3)	1·160 (3)	179·3 (3)	163·1 (3)	(i)
Ho(C₅H₄NCOOH)₃(H₂O)₂Cr(NCS)₆.2H₂O*	Cr³⁺	Octahedral	1·619 (7)	1·144 (7)	176·6 (7)	164·3 (6)	(j)
K₃Mo(NCS)₆.H₂O.CH₃COOH*	Mo³⁺	Octahedral	1·633 (8)	1·167 (9)	177·2 (7)	169·3 (5)	(k)

References: (a) Yamada *et al.* (1980); (b) Kendall & Lipscomb (1973); (c) Mereiter & Preisinger (1982); (d) Nieuwpoort & Verschoor (1981); (e) Engelfriet, Verschoor & den Brinker (1980); (f) Engelfriet, den Brinker, Verschoor & Gorter (1979); (g) Engelfriet, Verschoor & Vermin (1979); (h) Brattås *et al.* (1978); (i) Peterson *et al.* (1976); (j) Kay, Moore & Glick (1972); (k) Knox & Eriks (1968).

* The average of a parameter P_{av} is taken as $P_{av} = (\sum P_i)/n$, and its e.s.d. is estimated as $\sigma_{av}/n^{1/2}$ to a first approximation. Here σ_{av} refers to the average of the e.s.d.'s.
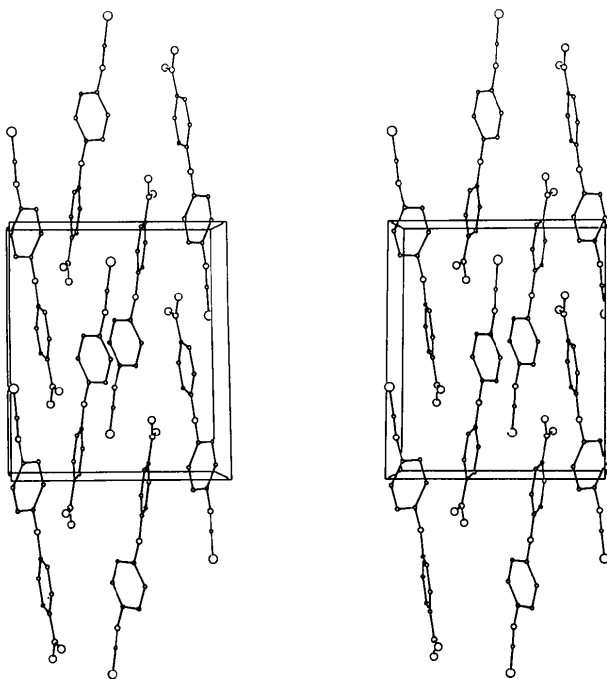

Fig. 2. Stereoscopic pair showing the [100] projection of the unit cell. The origin of the unit cell is in the lower-left-rear corner. The axial system is right-handed.

ing, 1960), explanations given for the deviation from linearity of these groups involve the crystal packing and variation in π-electron bonding in the N—X bonds (Nieuwpoort & Verschoor, 1981; Brattås, Jagner & Ljungström, 1978; Peterson, Von Dreele & Brown, 1976; Knox & Eriks, 1968). Of interest is the C—N—H

angle of 132 (2)° obtained from a microwave spectroscopy study for HNCS (Yamada, Winnewisser, Winnewisser, Szalanski & Gerry, 1980), which is expected to exhibit different resonance from those of metal complexes. The C—N distance in amoscanate [1·154 (3) Å] resembles those in metal complexes, but the S—C distance is closer to those of HNCS and B₁₀H₁₃NCS (Kendall & Lipscomb, 1973).

Fig. 2 shows a stereoscopic view of the [100] projection of the unit cell. The shortest intermolecular contact between non-H atoms is 3·203 (2) Å for N(2)···O(2), and that between non-H and H atoms is 2·43 (2) Å for O(2)···H(N2).

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Structure of Diphenacyl Selenide, C₁₆H₁₄O₂Se

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Abstract. $M_r = 317.2$, monoclinic, $P2_1/c$, $a = 5.618$ (3), $b = 9.963$ (5), $c = 25.235$ (7) Å, $\beta = 95.08$ (4)°, $V = 1406.9$ Å³, $Z = 4$, $D_x = 1.50$ Mg m⁻³, $\lambda(Cu\,K\alpha) = 1.54178$ Å, $\mu = 3.326$ mm⁻¹, $F(000) = 640$. Final $R = 0.071$ for 1932 independent reflections. Selenium forms two primary Se—C bonds and one secondary Se—O intramolecular contact.

Introduction. Diphenacyl selenide (R_2Se) is a symmetric selenide often encountered as an intermediate in SeO_2 oxidation and related reactions. Such carbon-bonded Se molecules play important roles in the metabolism and toxicity of selenium.

Experimental. R_2Se prepared by reduction of dichlorodiphenacylselenium(IV) by thiourea in acetone medium. Slow evaporation of ethyl acetate solution containing R_2Se yielded colourless, needle-shaped crystals; Weissenberg and precession photographs gave preliminary unit-cell dimensions and symmetry information; systematic absences fixed the space group to be $P2_1/c$; cell parameters were obtained by least-squares refinement of θ values of 25 high-angle reflections; crystal dimensions $0.26 \times 0.25 \times 0.08$ mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$ radiation, $\omega/2\theta$ scan mode, $2 < \theta < 78°$; 3350 reflections collected, out of which 1932