Table 2. *Bond distances* (Å) *and angles* (°)

C(2)–C(1)	1·548 (3)	C(13)–C(8)	1·409 (4)
C(5)–C(1)	1·547 (4)	C(10)–C(9)	1·397 (4)
C(17)–C(1)	1·504 (4)	C(14)–C(9)	1·490 (5)
C(3)–C(2)	1·525 (3)	C(11)–C(10)	1·378 (5)
C(6)–C(2)	1·518 (4)	C(12)–C(11)	1·391 (5)
C(4)–C(3)	1·522 (4)	C(15)–C(11)	1·502 (5)
C(5)–C(4)	1·523 (5)	C(13)–C(12)	1·355 (4)
C(7)–C(6)	1·530 (3)	C(16)–C(13)	1·509 (4)
S(1)–C(6)	1·831 (2)	O(1)–C(17)	1·312 (3)
C(8)–S(1)	1·777 (3)	O(2)–C(17)	1·219 (3)
C(9)–C(8)	1·408 (4)		
C(5)–C(1)–C(2)	102·1 (2)	C(13)–C(8)–C(9)	119·8 (2)
C(17)–C(1)–C(2)	113·3 (2)	C(10)–C(9)–C(8)	117·6 (3)
C(17)–C(1)–C(5)	110·2 (2)	C(14)–C(9)–C(8)	123·5 (3)
C(3)–C(2)–C(1)	104·6 (2)	C(14)–C(9)–C(10)	118·9 (3)
C(6)–C(2)–C(1)	115·0 (2)	C(11)–C(10)–C(9)	123·3 (3)
C(6)–C(2)–C(3)	116·0 (2)	C(12)–C(11)–C(10)	116·8 (3)
C(4)–C(3)–C(2)	107·3 (2)	C(15)–C(11)–C(10)	122·4 (3)
C(5)–C(4)–C(3)	106·5 (2)	C(12)–C(11)–C(15)	120·7 (3)
C(4)–C(5)–C(1)	105·8 (2)	C(11)–C(12)–C(13)	123·1 (3)
C(7)–C(6)–C(2)	113·0 (2)	C(8)–C(13)–C(12)	119·4 (3)
S(1)–C(6)–C(2)	108·1 (2)	C(8)–C(13)–C(16)	122·1 (3)
S(1)–C(6)–C(7)	111·4 (2)	C(12)–C(13)–C(16)	118·5 (3)
C(8)–S(1)–C(6)	101·5 (1)	C(1)–C(17)–O(1)	114·4 (2)
C(9)–C(8)–S(1)	119·9 (2)	C(1)–C(17)–O(2)	123·5 (2)
C(13)–C(8)–S(1)	120·4 (2)	O(1)–C(17)–O(2)	122·1 (2)

Related literature. The synthesis and crystal structure of the related 2-(1-phenylthioethyl)pent-3-ene-1-carboxylic acid has been described by Michel *et al.* (1980).

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Structure of 2-(2-Chloro-3-pyridylthio)-3,5,6-trimethylphenol at 163 K

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Abstract. $C_{14}H_{14}ClNOS$, $M_r = 279{\cdot}78$, monoclinic, $P2_1/c$, $a = 7{\cdot}5049\,(10)$, $b = 20{\cdot}487\,(3)$, $c = 8{\cdot}6131\,(10)$ Å, $\beta = 90{\cdot}527\,(8)°$, $V = 1324{\cdot}2\,(3)$ Å^3, $Z = 4$, $D_x(163\ \mathrm{K}) = 1{\cdot}403$, $D_m(295\ \mathrm{K}) = 1{\cdot}343$ g cm^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 4{\cdot}242$ cm^{-1}, $F(000) = 584$, $R = 0{\cdot}0407$ for 2930 observed reflections. The molecule is a precursor to 6,7,9-trimethyl-4-azaphenoxathiin through displacement of the chloro substituent through release of HCl. The angle between the aromatic rings is 87·52 (6)°; the C–S–C plane is nearly parallel to the pyridine plane [dihedral angle: 4·33 (9)°]. The O atom is 5·579 (2) Å from the Cl atom. Bond lengths are normal: C–S, 1·766, 1·774 Å; C–Cl, 1·744 Å; aromatic C–C, 1·370–1·407 Å; other C–C, 1·506–1·511 Å; C–N, 1·319, 1·342 Å. The C–S–C angle is 102·98 (8)°.

Experimental. A summary of data collection and structural refinement is given in Table 1. Density measured by flotation. S and Cl positions obtained from *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Electron density map afforded remaining non-H positions. Electron density difference map at $R = 0{\cdot}061$ provided H positions as peaks of 0·57–0·84 e Å^{-3}. All positions and thermal parameters (isotropic H atoms) refined by full-matrix

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Table 1. *Crystallographic summary for* $C_{14}H_{14}ClNOS$

(*A*) Data collection (163 K)^{i,ii}	
Radiation	Mo $K\alpha$
Mode	ω scan
Scan range	Symmetrically over 1·0° about $K\alpha_{1,2}$ maximum
Background	Offset 1·0 and −1·0° in ω from $K\alpha_{1,2}$ maximum
Scan rate (° min^{-1})	2·5–5·0
Exposure time (h)	57·6
Stability analysis	
Computed *s*	a = 0·00051 (18)
t	b = −0·000005 (3)
Correction range (on *I*)	0·987–1·000
2θ range (°)	4·0–60·0
Range in *hkl*, min.	0,0,−12
max.	10,28,12
Total reflections measured	3852
Data-crystal dimensions (mm)	0·45 × 0·4 × 0·2
Absorption correction	None applied
(*B*) Structure refinement^{iii}	
Instability factor, p^{ii}	0·04
Reflections used ($F \geq 4\sigma_F$)	2930
No. of variables	219
Goodness of fit, *S*	1·396
R, *wR*	0·0407, 0·0467
R for all data	0·0609
Max. shift/e.s.d.	0·0044
Max. density in difference map (e Å^{-3})	0·46 [between C(7) and C(8)]
Min. density in difference map (e Å^{-3})	−0·23

Notes: (i) Unit-cell parameters were obtained by least-squares refinement of the setting angles of 45 reflections with $21{\cdot}3 < 2\theta < 30{\cdot}1°$. (ii) Syntex $P2_1$ autodiffractometer with a graphite monochromator and a Syntex LT-1 inert-gas (N_2) low-temperature delivery system. Data reduction was carried out as described by Riley & Davis (1976). Crystal and instrument stability were monitored by re-measurement of four check reflections after every 96 reflections. As detailed by Henslee & Davis (1975), these data were analyzed to relate intensity to exposure time by the equation $y = 1{\cdot}0 + sx + tx^2$ where x is exposure time (h), y is fractional intensity relative to $x = 0$ and s and t are coefficients determined by least-squares fit. (iii) Function minimized was $\sum w(F_o - F_c)^2$, where $w = \sigma_F^{-2}$.

least squares (*SHELX*76, Sheldrick, 1976). Final parameters are in Table 2.* Scattering factors and anomalous-dispersion corrections for S, Cl, O, N and C from *International Tables for X-ray Crystallography* (1974); scattering factors for H from Stewart, Davidson & Simpson (1965). Least-squares-planes' program from Cordes (1983); others cited by Gadol & Davis (1982).

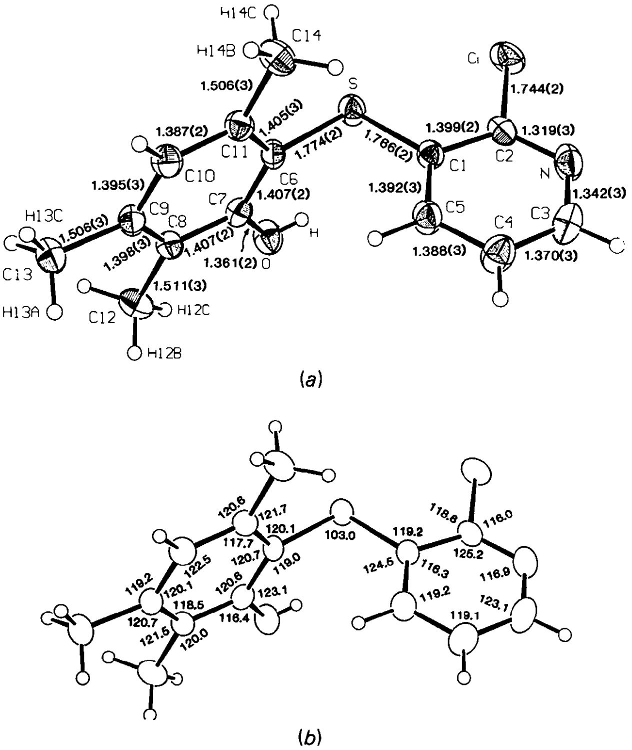
Fig. 1 shows the atom labeling, bond lengths and angles, Fig. 2 the molecular packing.

Related literature. For work on S-bridged aromatic rings, see: Puig-Torres, Martin, Larson & Simonsen (1984); Kimura, Simonsen, Caldwell & Martin (1981); Cody & Lehmann (1982); Korp, Bernal & Martin (1981); von Deuten & Klar (1981); Andreetti, Garbarczyk & Krolikowska (1981).

Table 2. *Positional parameters and* U_{eq}/U *for* $C_{14}H_{14}ClNOS$

	x	y	z	U_{eq}*/U (Å^2)
Cl	0·77362 (6)	0·06476 (3)	0·02602 (7)	0·0402 (2)
S	0·45736 (6)	0·12183 (2)	−0·16431 (5)	0·02809 (13)
O	0·1568 (2)	0·05394 (7)	−0·3396 (2)	0·0345 (4)
N	0·5536 (2)	0·04299 (8)	0·2527 (2)	0·0355 (5)
C(1)	0·4223 (2)	0·09530 (8)	0·0281 (2)	0·0235 (5)
C(2)	0·5643 (2)	0·06791 (8)	0·1119 (2)	0·0263 (5)
C(3)	0·3942 (3)	0·04576 (11)	0·3218 (2)	0·0384 (6)
C(4)	0·2463 (3)	0·07271 (11)	0·2528 (2)	0·0361 (6)
C(5)	0·2594 (3)	0·09789 (10)	0·1037 (2)	0·0309 (5)
C(6)	0·2482 (2)	0·15584 (9)	−0·2176 (2)	0·0238 (5)
C(7)	0·1277 (2)	0·11785 (9)	−0·3051 (2)	0·0250 (5)
C(8)	−0·0312 (2)	0·14531 (9)	−0·3636 (2)	0·0259 (5)
C(9)	−0·0678 (2)	0·21070 (9)	−0·3300 (2)	0·0269 (5)
C(10)	0·0514 (2)	0·24710 (9)	−0·2398 (2)	0·0275 (5)
C(11)	0·2102 (2)	0·22142 (9)	−0·1834 (2)	0·0253 (5)
C(12)	−0·1574 (3)	0·10434 (12)	−0·4603 (3)	0·0360 (6)
C(13)	−0·2354 (3)	0·24264 (12)	−0·3902 (3)	0·0366 (6)
C(14)	0·3364 (3)	0·26290 (11)	−0·0890 (3)	0·0340 (6)
H	0·239 (4)	0·043 (2)	−0·303 (4)	0·066 (10)
H(3)	0·392 (3)	0·0288 (11)	0·420 (3)	0·042 (6)
H(4)	0·143 (3)	0·0734 (12)	0·306 (3)	0·049 (7)
H(5)	0·154 (3)	0·1163 (11)	0·049 (2)	0·035 (6)
H(10)	0·019 (3)	0·2913 (11)	−0·213 (3)	0·036 (6)
H(12)*A*	−0·178 (4)	0·1274 (14)	−0·567 (3)	0·065 (8)
H(12*B*	−0·266 (4)	0·0956 (15)	−0·409 (4)	0·072 (9)
H(12*C*	−0·112 (4)	0·0613 (14)	−0·481 (3)	0·068 (9)
H(13)*A*	−0·334 (4)	0·2201 (13)	−0·354 (3)	0·054 (7)
H(13*B*	−0·239 (3)	0·2460 (12)	−0·499 (3)	0·049 (7)
H(13)*C*	−0·249 (4)	0·289 (2)	−0·349 (4)	0·078 (10)
H(14)*A*	0·374 (3)	0·2408 (13)	0·006 (3)	0·059 (8)
H(14)*B*	0·276 (4)	0·3039 (14)	−0·056 (3)	0·059 (8)
H(14)*C*	0·434 (4)	0·271 (2)	−0·148 (4)	0·082 (10)

* For non-H atoms, the U value is U_{eq}, calculated as $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{A}_{ij}$ where $\mathbf{A}_{ij}$ is the dot product of the *i*th and *j*th direct-space unit-cell vectors.



(*a*)

(*b*)

Fig. 1. (*a*) View of the molecule showing atom labeling and bond lengths (Å). Ellipsoids scaled to 50% probability level. (*b*) Bond angles (°); e.s.d.'s are 0·2–0·3°.

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* Tables of anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles, least-squares planes and structure-factor amplitudes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42751 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

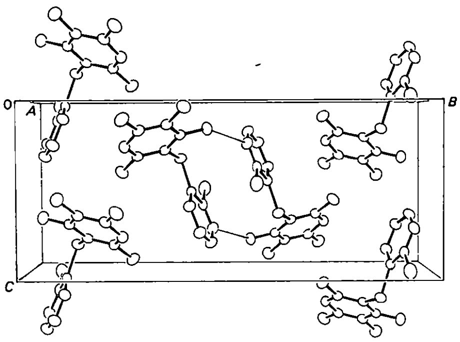

Fig. 2. Molecular packing as viewed along the *c* axis. Centrosymmetric pairs are joined through weak hydrogen bonds [N···H, 2·39 (3) Å]. All other intermolecular contacts are greater than the sum of the relevant van der Waals radii.

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