$V = 798.5\,(3)$ Å^3
$Z = 2$
$D_x = 1.183$ Mg m^{-3}

0.25 × 0.20 × 0.20 mm
Colourless

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Data collection

Rigaku AFC-6S diffractometer
2θ scans
Absorption correction:
    ψ scan (Coppens, Leiserowitz & Rabinovich, 1965)
$T_{min} = 0.953$, $T_{max} = 1.000$
1513 measured reflections
1407 independent reflections
1213 observed reflections
    $[F \geq 4\sigma(F)]$

$R_{int} < 0.001$
$\theta_{max} = 25°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 10$
$l = -12 \rightarrow 11$
3 standard reflections
    monitored every 150
    reflections
    intensity decay: <3%

Refinement

Refinement on F
$R = 0.0563$
$wR = 0.0534$
$S = 0.5$
1213 reflections
139 parameters
All H-atom parameters refined
Unit weights applied

$(\Delta/\sigma)_{max} = 0.012$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Acta Cryst. (1996). C52, 1239–1241

[3-(4-Chlorophenyl)-5-methylthio-4,5-dihydro-5-isoxazolyl]acetonitrile

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Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O(1)	0.8080 (3)	−0.1612 (2)	0.6447 (2)	0.075 (1)
O(2)	0.6246 (3)	0.1100 (3)	0.3325 (2)	0.106 (1)
N	0.7549 (2)	0.0715 (2)	0.5420 (2)	0.039 (1)
C(1)	0.9253 (3)	0.4701 (3)	0.5111 (3)	0.048 (1)
C(2)	0.9186 (3)	0.2947 (3)	0.5145 (3)	0.048 (1)
C(3)	0.7773 (3)	0.2408 (3)	0.5523 (3)	0.043 (1)
C(4)	0.8221 (3)	−0.0235 (3)	0.6494 (2)	0.045 (1)
C(5)	0.9132 (5)	0.0517 (4)	0.7731 (3)	0.075 (1)
C(6)	0.6668 (3)	0.0173 (3)	0.4192 (3)	0.055 (1)
C(7)	0.6250 (4)	−0.1486 (3)	0.3977 (4)	0.068 (1)

Table 2. *Selected torsion angles (°)*

C(1^i)—C(1)—C(2)—C(3)	173.4 (3)	C(3)—N—C(6)—O(2)	3.7 (4)
C(1)—C(2)—C(3)—N	173.3 (2)	C(3)—N—C(4)—O(1)	−179.3 (3)
C(4)—N—C(3)—C(2)	90.6 (3)		

Symmetry code: (i) $2 - x$, $1 - y$, $1 - z$.

The structure was refined by full-matrix least squares using *SHELXTL-Plus* (Sheldrick, 1990). Data collection, all calculations and graphics were carried out using *SHELXTL-Plus*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: VJ1033). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Abstract

The title compound, $C_{12}H_{11}ClN_2OS$, is formed by the reaction between 6-(4-chlorophenyl)-4-methylthio-2-oxo-2H-pyran-3-carbonitrile and hydroxylamine hydrochloride in pyridine solution. The best plane through the five atoms of the isoxazole ring makes an angle of 10.48 (8)° with the plane of the aromatic ring.

Comment

The high antispasmodic activity of isoxazoles (Naruto *et al.*, 1982, 1983) has prompted us to synthesize different types of isoxazoles and their derivatives for structure–activity studies. The reaction of 6-(4-chlorophenyl)-4-methylthio-2-oxo-2H-pyran-3-carbonitrile, (I), with hydroxylamine hydrochloride in pyridine has recently been reported to give three compounds, (II), (III) and (IV), each containing an isoxazole moiety (Ram, Hussaini, Singh & Shoeb, 1993); compound (III) was obtained only as a gummy mass. We now report a modified preparative procedure which gives (III) as a crystalline solid. The X-ray structure of (III) was determined in order to obtain an unambiguous characterization.

(I)

(II)

(III)

(IV)
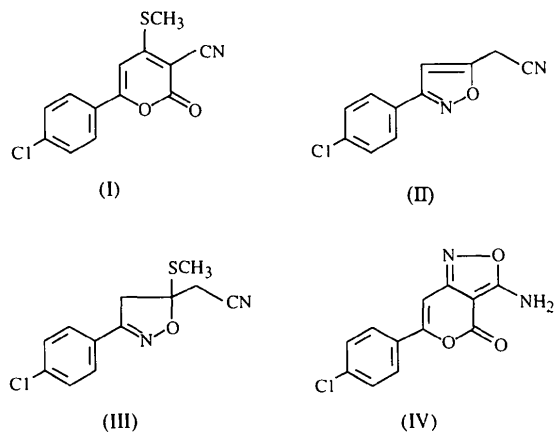
The molecular structure of (III) is represented in Fig. 1. All of the bond lengths and angles are unexceptional and, in particular, the isoxazole ring dimensions are very similar to those reported earlier (Blake, Gould, Paton & Young, 1993; Houk, Duh, Wu & Moses, 1986). The best plane through the five atoms of the isoxazole ring has an r.m.s. deviation of 0.025 Å and this plane is inclined at an angle of 10.48 (8)° to the plane of the aromatic ring. This lack of planarity between the two ring systems is also evident in the torsion angles across the C4—C7 bond [e.g. C3—C4—C7—N1 169.96 (15)°]. The acetonitrile group is linear within experimental error.
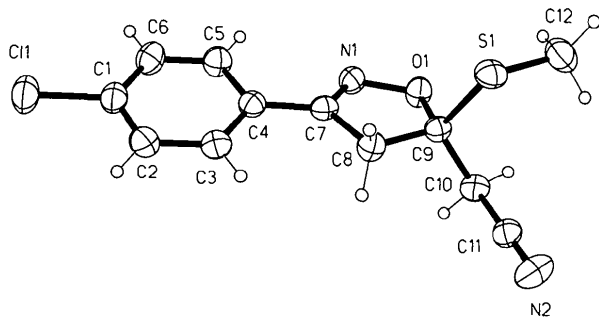

Fig. 1. View of the title molecule showing the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% probability level.

The origin of the intermolecular packing forces is not obvious. The aromatic rings stack in parallel but are not exactly superimposed. Furthermore, there is a centre of inversion between adjacent molecules so that the overall packing situation involves a 'head-to-tail' alignment of molecules.

Experimental

For the preparation of (III), 6-(4-chlorophenyl)-4-methyl-thio-2-oxo-2H-pyran-3-carbonitrile [(I); 1.665 g, 6 mmol] was dissolved in pyridine (15 ml) at 343–353 K. Hydroxylamine hydrochloride (486 mg, 7 mmol) was added and the reaction

mixture was stirred for 40 h at 343–353 K. The procedure previously described by Ram, Hussaini, Singh & Shoeb (1993) involved refluxing conditions. Pyridine was distilled off under reduced pressure and the residue was taken up in ethyl acetate, washed with water and dilute HCl, and dried over Na_2SO_4. The gummy mass obtained on removal of ethyl acetate was subjected to column chromatography on silica gel, whereupon compounds (II) and (III) eluted with ethyl acetate–petroleum ether (12% v/v). Compound (III) crystallized from a chloroform–petroleum ether solution as white crystals (m.p. 361–362 K).

Crystal data

$C_{12}H_{11}ClN_2OS$	Mo $K\alpha$ radiation
$M_r = 266.74$	$\lambda = 0.71073$ Å
Monoclinic	Cell parameters from 31
$P2_1/c$	reflections
$a = 14.157 (9)$ Å	$\theta = 10–11.5°$
$b = 9.891 (6)$ Å	$\mu = 0.457$ mm^{-1}
$c = 9.312 (6)$ Å	$T = 210 (2)$ K
$\beta = 106.85 (5)°$	Block
$V = 1247.9 (14)$ Å^3	$0.51 \times 0.36 \times 0.25$ mm
$Z = 4$	Colourless
$D_x = 1.420$ Mg m^{-3}	
D_m not measured	

Data collection

Siemens $P3/R3$ diffractometer	$R_{int} = 0.0162$
eter	$\theta_{max} = 25.05°$
ω–2θ scans	$h = -16 \rightarrow 16$
Absorption correction:	$k = -11 \rightarrow 1$
analytical (Alcock & Marks, 1993)	$l = 0 \rightarrow 11$
$T_{min} = 0.83$, $T_{max} = 0.90$	3 standard reflections monitored every 200
2643 measured reflections	reflections
2204 independent reflections	intensity decay: 5%
1951 observed reflections	
$[I > 2\sigma(I)]$	

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.190$ e Å^{-3}
$R[F^2 > 2\sigma(F^2)] = 0.0304$	$\Delta\rho_{min} = -0.223$ e Å^{-3}
$wR(F^2) = 0.0897$	Extinction correction:
$S = 1.041$	SHELXL93 (Sheldrick,
2199 reflections	1993)
156 parameters	Extinction coefficient:
H atoms riding	0.0568 (31)
$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2 + 0.4474P]$	Atomic scattering factors from International Tables
where $P = (F_o^2 + 2F_c^2)/3$	for Crystallography (1992,
$(\Delta/\sigma)_{max} = 0.001$	Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cl1	0.50564 (3)	1.05307 (6)	-0.82771 (5)	0.0505 (2)
S1	0.76711 (3)	0.95491 (4)	0.18839 (5)	0.0359 (2)
O1	0.86082 (8)	1.09164 (11)	0.02141 (12)	0.0336 (3)
N1	0.80618 (10)	1.12682 (13)	-0.12658 (14)	0.0331 (3)
N2	0.92218 (11)	0.6395 (2)	0.1991 (2)	0.0495 (4)

C1	0.57869(12)	1.0458(2)	−0.6416(2)	0.0369(4)
C2	0.59545(12)	0.9228(2)	−0.5713(2)	0.0392(4)
C3	0.65336(12)	0.9165(2)	−0.4232(2)	0.0365(4)
C4	0.69457(11)	1.0330(2)	−0.3472(2)	0.0304(3)
C5	0.67570(12)	1.1566(2)	−0.4220(2)	0.0377(4)
C6	0.61792(13)	1.1633(2)	−0.5688(2)	0.0406(4)
C7	0.75666(11)	1.0249(2)	−0.1909(2)	0.0291(3)
C8	0.76964(12)	0.9013(2)	−0.0948(2)	0.0324(4)
C9	0.83692(11)	0.95473(15)	0.0535(2)	0.0283(3)
C10	0.93677(11)	0.8840(2)	0.1088(2)	0.0318(4)
C11	0.92848(11)	0.7468(2)	0.1598(2)	0.0334(4)
C12	0.85166(14)	1.0292(2)	0.3512(2)	0.0473(5)

Table 2. *Selected geometric parameters (Å, °)*

C11—C1	1.743(2)	N2—C11	1.135(2)
S1—C12	1.794(2)	C7—C8	1.495(2)
S1—C9	1.810(2)	C8—C9	1.527(2)
O1—N1	1.414(2)	C9—C10	1.527(2)
O1—C9	1.448(2)	C10—C11	1.454(2)
N1—C7	1.274(2)		
C12—S1—C9	102.83(9)	O1—C9—C8	105.04(12)
N1—O1—C9	109.66(11)	C8—C9—C10	114.88(14)
C7—N1—O1	109.54(13)	C10—C9—S1	113.86(11)
N1—C7—C4	120.82(14)	C11—C10—C9	112.54(13)
N1—C7—C8	114.23(14)	N2—C11—C10	179.7(2)
C7—C8—C9	101.21(13)		
O1—N1—C7—C8	0.2(2)	C3—C4—C7—C8	−9.0(2)
C3—C4—C7—N1	169.96(15)	N1—C7—C8—C9	3.3(2)
C5—C4—C7—N1	−10.1(2)	C7—C8—C9—O1	−5.21(15)

The temperature of the crystal was controlled using an Oxford Cryosystem Cryostream Cooler (Cosier & Glazer, 1986). H atoms were added in calculated positions and refined using a riding model. Anisotropic displacement parameters were used for all non-H atoms, while H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the atom to which they are attached.

Data collection: Siemens *P3R3* system. Cell refinement: Siemens *P3R3* system. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1060). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Redetermination of Octahydrochrysene

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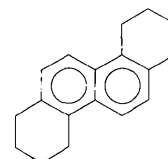
Abstract

The central rings of 1,2,3,4,7,8,9,10-octahydrochrysene, $C_{18}H_{20}$, are essentially planar, with the r.m.s. deviation of the atoms defining the plane from the best-fit plane being 0.013(2) Å. The outer rings are found to be substantially non-planar, contrary to the conclusion of an earlier study based on photographic data [Ferrier & Iball (1958). *Acta Cryst.* **11**, 325–329]. The C—C single-bond distances in the outer rings are quite uniform, with the range of observed values varying by only 0.017(6) Å. There are no notably close intermolecular approaches.

Comment

Octahydrochrysene, (I), is of interest as a potential host crystalline material for magnetic or optical studies of substituted guest molecules. A previous structural analysis by Ferrier & Iball (1958) based on two-dimensional Fourier methods and with fixed H-atom geometry resulted in *R* values of 0.15 and 0.16 for the

(I)