

144.6° again [C(7), O(3), C(8)] (*cf.* *cis*-'benzene trioxide' 106.9°). The dihedral angle of the three open triangles C(2)–C(3)–C(4), C(5)–C(6)–C(7) and C(8)–C(9)–C(1) is 106.7°. The distances of the *anti* protons H(4), H(8), H(12) are 2.25 and 2.31 Å, which are slightly shorter than the van der Waals distance (2.4 Å). The steric pressure results in a diminution of the H–C–H bond angles to 105 and 104°.

As can be seen in Fig. 2, which shows the contents of the unit cell, there are merely van der Waals forces between the molecules. The shortest intermolecular distance between H(3) and O(1) is 2.68 Å, equal to the van der Waals distance (2.6 Å).

Most of the calculations were performed with the XRAY 70 system of crystallographic programs (Stewart, Kundell & Baldwin, 1970) on the Univac 1100/80 computer of the Rechenzentrum Freiburg. Financial help of the Deutsche Forschungsgemeinschaft is gratefully acknowledged.

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## The Structure of 5-(*N*-Methylthiocarbamoylimino)-4-phenyl-3-phenylamino-4*H*-1,2,4-thiadiazoline

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**Abstract.** C<sub>16</sub>H<sub>15</sub>N<sub>5</sub>S<sub>2</sub>, monoclinic,  $P2_1/c$ ,  $a = 9.487$  (6),  $b = 17.447$  (9),  $c = 10.063$  (5) Å,  $\beta = 103.15$  (4)°,  $M_r = 341.45$ ,  $D_c = 1.398$  Mg m<sup>-3</sup>,  $Z = 4$ ,  $\mu(\text{Mo } K\alpha) = 0.285$  mm<sup>-1</sup>, the final  $R$  is 0.0761 for a total of 1051 independent reflections. The 1:1 adduct of Hector's base and methyl isothiocyanate forms without a heterocyclic rearrangement: the compound is a 1,2,4-thiadiazoline in which S–N is 1.697 (10) and S...S is 2.822 (5) Å.

**Introduction.** The adduct was prepared by reaction of Hector's base with methyl isothiocyanate in ethanol: crystals were grown from ethanol–acetone solution. The space group was uniquely assigned from the systematic absences. The intensities of 1405 unique reflections having  $0^\circ \leq \theta \leq 30^\circ$  in the quadrant  $\pm h, +k, +l$  ( $k = 0-20$ ;  $0.000^\circ \leq \mu \leq 24.038^\circ$ ) were measured in the  $\omega-2\theta$  scan mode using a Stoe

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STADI-2 diffractometer, with a scan speed of 0.01° s<sup>-1</sup> in  $\omega$  and a scan width of 1.0° in  $\omega$ : backgrounds were measured at each end of the scan for a time of (scan time/2). Standard reflections, measured after every 50 reflections, showed only small random deviations from their means. Lorentz and polarization but no absorption corrections were applied.

The structure was solved by direct methods using *SHELX 76* (Sheldrick, 1976) and refined by blocked full-matrix least-squares with complex neutral-atom scattering factors (Cromer & Mann, 1968; Cromer & Liberman, 1970) for 1051 data having  $F_o > 6\sigma(F_o)$ . With anisotropic temperature factors for S, C, N, a common isotropic temperature factor for phenyl H atoms in calculated positions, a second isotropic temperature factor for H(1) bonded to N(3), and interlayer scale factors refined, the refinement converged to  $R = \sum \Delta / \sum F_o = 0.0740$  and  $R_G = (\sum w\Delta^2 / \sum wF_o^2)^{1/2} = 0.0825$ . One of the C atoms, C(8), was marginally but persistently non-positive definite: when this atom was

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refined isotropically, the refinement converged to  $R = 0.0761$  and  $R_G = 0.0843$ . When weights  $w = 1/\sigma^2(F)$  were introduced, C(8) remained non-positive definite, and the geometry of the phenyl rings was rather less satisfactory: all results quoted are for unit weights and C(8) isotropic. The H attached to N(5) was not located. The final coordinates are listed in Table 1, bond distances in Table 2 and bond angles in Table 3. Fig. 1 shows the molecule, and the numbering scheme for the atoms and bonds.\*

\* 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 35938 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates ( $\times 10^4$ ; for H  $\times 10^3$ ) and equivalent isotropic temperature factors ( $\text{\AA}^2 \times 10^3$ )

The equivalent isotropic temperature factor is defined as the geometric mean of the diagonal components of the diagonalized matrix of  $U_{ij}$ .

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}/U$
S(1)	4737 (4)	1139 (2)	-1071 (3)	27 (2)
S(2)	2890 (3)	-66 (2)	-2254 (3)	32 (2)
N(1)	5735 (11)	1843 (6)	-137 (9)	26 (6)
N(2)	4244 (9)	1456 (5)	1237 (8)	14 (5)
N(3)	5989 (11)	2429 (7)	2026 (11)	24 (7)
N(4)	2708 (10)	472 (6)	218 (9)	23 (6)
N(5)	1218 (11)	-480 (6)	-589 (10)	32 (7)
C(1)	5378 (14)	1924 (7)	1031 (13)	23 (8)
C(2)	3744 (12)	939 (7)	160 (12)	19 (7)
C(3)	2235 (12)	-19 (8)	-838 (12)	27 (7)
C(4)	514 (16)	-1096 (9)	-1557 (15)	36 (10)
C(5)	3719 (12)	1427 (7)	2481 (10)	10 (7)
C(6)	3897 (13)	766 (8)	3237 (12)	37 (9)
C(7)	3385 (15)	747 (9)	4436 (13)	40 (10)
C(8)	2700 (13)	1361 (7)	4806 (13)	34 (3)*
C(9)	2524 (13)	2017 (8)	4065 (13)	28 (8)
C(10)	3048 (13)	2058 (7)	2851 (12)	31 (8)
C(11)	7158 (13)	2915 (7)	1881 (11)	23 (8)
C(12)	8346 (14)	2996 (8)	2977 (13)	37 (8)
C(13)	9449 (14)	3476 (8)	2884 (14)	40 (9)
C(14)	9392 (15)	3887 (9)	1691 (15)	40 (10)
C(15)	8210 (17)	3815 (8)	613 (14)	46 (9)
C(16)	7041 (14)	3354 (8)	719 (14)	38 (9)
H(1)	558 (14)	247 (8)	262 (14)	54 (42)*

\* Atom refined isotropically, see text.

Table 2. Bond distances ( $\text{\AA}$ )

<i>a</i>	1.697 (10)	<i>h</i>	1.323 (17)	<i>o</i>	1.349 (20)	<i>v</i>	1.361 (20)
<i>b</i>	1.303 (17)	<i>i</i>	1.502 (18)	<i>p</i>	1.356 (19)	<i>w</i>	1.389 (21)
<i>c</i>	1.403 (16)	<i>j</i>	1.681 (13)	<i>q</i>	1.422 (19)	<i>x</i>	1.377 (19)
<i>d</i>	1.407 (14)	<i>k</i>	2.822 (5)	<i>r</i>	1.365 (18)	<i>y</i>	1.394 (21)
<i>e</i>	1.754 (13)	<i>l</i>	1.450 (15)	<i>s</i>	1.361 (16)	<i>z</i>	1.381 (18)
<i>f</i>	1.288 (15)	<i>m</i>	1.370 (18)	<i>t</i>	1.430 (17)		
<i>g</i>	1.360 (15)	<i>n</i>	1.400 (20)	<i>u</i>	1.393 (16)		
		N(3)—H(1)	0.79 (15)				

Table 3. Bond angles ( $^\circ$ )

<i>ab</i>	110.6 (9)	<i>ek</i>	76.6 (4)	<i>op</i>	121.8 (14)
<i>ae</i>	94.3 (5)	<i>fg</i>	118.8 (10)	<i>pq</i>	119.4 (13)
<i>ak</i>	170.8 (4)	<i>gh</i>	111.1 (11)	<i>qr</i>	118.0 (12)
<i>bc</i>	115.8 (10)	<i>gj</i>	125.0 (10)	<i>st</i>	121.3 (11)
<i>bs</i>	125.4 (12)	<i>hi</i>	123.2 (11)	<i>tu</i>	119.1 (11)
<i>cd</i>	113.3 (10)	<i>hj</i>	123.9 (9)	<i>tz</i>	120.0 (10)
<i>cl</i>	125.0 (9)	<i>jk</i>	85.4 (4)	<i>uw</i>	120.3 (12)
<i>cs</i>	118.7 (12)	<i>lm</i>	119.1 (11)	<i>uz</i>	120.6 (12)
<i>de</i>	105.9 (8)	<i>lr</i>	118.7 (11)	<i>vw</i>	119.8 (12)
<i>df</i>	119.8 (11)	<i>mn</i>	118.4 (13)	<i>wx</i>	119.9 (14)
<i>dl</i>	121.4 (9)	<i>mr</i>	122.2 (12)	<i>xy</i>	120.8 (13)
<i>ef</i>	134.2 (9)	<i>no</i>	120.1 (13)	<i>yz</i>	118.2 (11)
		H(1)—N(3)—C(1)	115 (10)		
		H(1)—N(3)—C(11)	124 (10)		

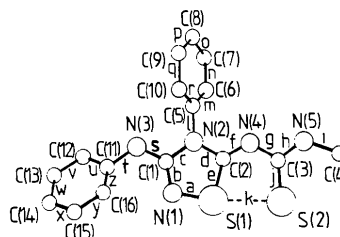
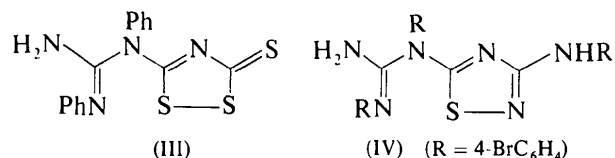
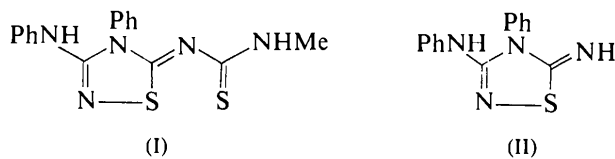


Fig. 1. The molecule showing the numbering of the atoms and bonds.

**Discussion.** The structure consists of molecules of constitution (I), in which simple addition of methyl isothiocyanate to Hector's base (II) (Butler, Glidewell & Liles, 1978a) has occurred. This is in marked contrast to the addition of carbon disulphide to Hector's base to give (III) (Butler, Glidewell & Liles, 1978b), and of 4-bromophenylcyanamide to the 4-bromophenyl analogue of Hector's base to give (IV) (Akiba, Tsuchiya, Inamoto, Onuma, Nagashima & Nakamura, 1976).



The dimensions of the heterocyclic ring are similar to those of the other six 1,2,4-thiadiazolines whose structures have been reported (Glidewell & Liles, 1981;

Cuthbertson, Glidewell, Holden & Liles, 1979), and almost identical to those in Hector's base (II) itself (Butler, Glidewell & Liles, 1978a).

The S...S distance 2.822 (5) Å is substantially longer than those found in compounds containing a hypervalent S atom in the centre of an SSN chain, where the range of S-S is 2.435–2.497 Å (see Glidewell, Holden & Liles, 1980).

The intermolecular distance between H(1) and N(1<sup>i</sup>)\* is 2.53 Å, and may represent a weak hydrogen bond, with N(3)–H(1)···N(1<sup>i</sup>) of 140°. No other significant short inter- or intramolecular contacts occur.

Hector's base itself (II) and its adducts with carbon disulphide (III) and methyl isothiocyanate (I) all exhibit the same structure in the solid state as in solution, as determined by <sup>13</sup>C and <sup>15</sup>N NMR (Butler, Glidewell, Hussain & Maw, 1980): no prototropic shifts occur.

\* The superscript i refers to the symmetry position  $x, \frac{1}{2} - y, \frac{1}{2} + z$ .

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## 1,16-Dihydrodiindeno[5,4-c:4',5'-g]phenanthrene

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**Abstract.** C<sub>28</sub>H<sub>18</sub>, orthorhombic, *Pbcn*,  $a = 8.851$  (3),  $b = 15.722$  (5),  $c = 13.307$  (4) Å,  $M_r = 354.46$ ,  $V = 1851.7$  Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.27$  (1) (neutral buoyancy in aqueous ZnI<sub>2</sub>),  $D_c = 1.271$  Mg m<sup>-3</sup>,  $F(000) = 744$ ; Mo  $K\alpha$  radiation ( $\lambda_{\text{vac}} = 0.71073$  Å,  $\mu = 0.065$  mm<sup>-1</sup>). The structure was solved by direct methods and refined by full-matrix least squares to  $R = 0.068$  using 715 diffractometer reflections with  $F_o > 4\sigma(F_o)$ . The two arms of the helix are splayed further apart than in similar hexahelicenes and heptahelicenes, the dihedral angle between terminal-ring planes being 69.1°. Each molecule possesses a crystallographically required twofold axis.

**Introduction.** The structure determination reported here was undertaken so as to characterize unambiguously the title compound, (I), synthesized by Pesti & Katz (1980, unpublished work). The crystal structures of various helicenes have been reported (Lightner, Hefelfinger, Powers, Frank & Trueblood, 1972; Frank, Hefelfinger & Lightner, 1973; de Rango, Tsoucaris, Declercq, Germain & Putzeys, 1973; van den Hark &

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Noordik, 1973; van den Hark, Noordik & Beurskens, 1974; Beurskens, Beurskens & van den Hark, 1976; van den Hark & Beurskens, 1976; Le Bas, Navaza, Mauguén & de Rango, 1976; Le Bas, Navaza, Knossow & de Rango, 1976; Lindner & Kitschke, 1979; Marsh & Dunitz, 1979; Konno, Saito, Yamada & Kawazura, 1980) and a theoretical study has appeared (Navaza, Tsoucaris, Le Bas & de Rango, 1979).

