C(1)	1.3990 (8)	−0.0975 (5)	0.662 (2)	7.2 (4)
C(2)	1.3382 (7)	−0.0254 (4)	0.726 (2)	6.2 (3)
C(3)	1.2566 (6)	−0.0390 (4)	0.937 (1)	4.9 (3)
C(4)	1.2090 (6)	0.0356 (4)	1.018 (1)	4.6 (2)
C(5)	1.0794 (4)	0.1168 (3)	0.8586 (9)	3.5 (2)
C(6)	0.9996 (5)	0.1265 (3)	0.669 (1)	3.9 (2)
C(7)	0.9438 (5)	0.1915 (3)	0.6890 (9)	3.8 (2)
C(8)	0.9665 (4)	0.2499 (3)	0.8967 (8)	3.0 (2)
C(9)	1.0463 (4)	0.2408 (3)	1.081 (1)	3.6 (2)
C(10)	1.1020 (5)	0.1752 (3)	1.067 (1)	3.9 (2)
C(11)	0.9052 (4)	0.3224 (3)	0.9180 (9)	3.3 (2)
C(12)	0.8675 (5)	0.4660 (3)	0.736 (1)	3.8 (2)
C(13)	0.7307 (4)	0.4373 (3)	0.7531 (9)	3.0 (2)
C(14)	0.7002 (6)	0.3828 (3)	0.965 (1)	4.1 (2)
C(15)	0.6748 (5)	0.5131 (3)	0.786 (1)	3.9 (2)
C(16)	0.6834 (5)	0.5633 (3)	0.565 (1)	4.0 (2)
C(17)	0.6288 (5)	0.6390 (4)	0.596 (1)	4.2 (2)
C(18)	0.6474 (7)	0.6916 (4)	0.385 (1)	5.5 (3)
C(19)	0.593 (1)	0.7666 (5)	0.411 (2)	7.9 (5)

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

S(1)—S(2)	3.008 (2)	C(6)—C(7)	1.368 (6)
S(1)—C(11)	1.803 (4)	C(7)—C(8)	1.387 (6)
S(1)—C(12)	1.806 (5)	C(8)—C(9)	1.365 (6)
S(2)—C(11)	1.800 (5)	C(8)—C(11)	1.519 (6)
S(2)—C(14)	1.802 (5)	C(9)—C(10)	1.376 (6)
O(1)—C(4)	1.443 (6)	C(12)—C(13)	1.511 (6)
O(1)—C(5)	1.356 (5)	C(13)—C(14)	1.522 (6)
C(1)—C(2)	1.531 (8)	C(13)—C(15)	1.532 (6)
C(2)—C(3)	1.491 (8)	C(15)—C(16)	1.512 (7)
C(3)—C(4)	1.496 (7)	C(16)—C(17)	1.520 (6)
C(5)—C(6)	1.388 (6)	C(17)—C(18)	1.491 (7)
C(5)—C(10)	1.389 (6)	C(18)—C(19)	1.508 (13)
C(11)—S(1)—C(12)	100.3 (2)	C(8)—C(9)—C(10)	121.9 (5)
C(11)—S(2)—C(14)	99.1 (2)	C(5)—C(10)—C(9)	119.9 (5)
C(4)—O(1)—C(5)	118.1 (4)	S(1)—C(11)—S(2)	113.2 (3)
C(1)—C(2)—C(3)	113.3 (6)	S(1)—C(11)—C(8)	109.1 (3)
C(2)—C(3)—C(4)	113.1 (5)	S(2)—C(11)—C(8)	110.4 (3)
O(1)—C(4)—C(3)	109.3 (5)	S(1)—C(12)—C(13)	116.7 (4)
O(1)—C(5)—C(6)	116.2 (4)	C(12)—C(13)—C(14)	110.6 (4)
O(1)—C(5)—C(10)	125.4 (4)	C(12)—C(13)—C(15)	110.1 (4)
C(6)—C(5)—C(10)	118.3 (4)	C(14)—C(13)—C(15)	111.0 (4)
C(5)—C(6)—C(7)	120.7 (5)	S(2)—C(14)—C(13)	115.8 (3)
C(6)—C(7)—C(8)	121.0 (5)	C(13)—C(15)—C(16)	114.6 (4)
C(7)—C(8)—C(9)	118.2 (4)	C(15)—C(16)—C(17)	114.9 (4)
C(7)—C(8)—C(11)	121.0 (4)	C(16)—C(17)—C(18)	114.1 (4)
C(9)—C(8)—C(11)	120.8 (4)	C(17)—C(18)—C(19)	115.1 (6)

Azimuthal scans of several reflections revealed no need for an absorption correction; intensities were corrected for Lorentz-polarization effects. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984). The non-H atoms were refined with anisotropic displacement parameters. H atoms located by difference Fourier synthesis were refined with isotropic displacement parameters. Full-matrix least-squares refinement minimized $\Sigma w(|F_o|-|F_c|)^2$. All calculations were performed using the *TEXSAN* crystallographic software package (Molecular Structure Corporation, 1985).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71765 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1072]

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Anomalous Products Formed from *N*-(5,6-Dihydrobenzo[*h*]quinazolin-4-yl)-amidines and Hydroxylamine Hydrochloride

TAKASHI HIROTA,* KENJI SASAKI AND HIROSHI YAMAMOTO

Faculty of Pharmaceutical Sciences, Okayama University, Tsushima, Okayama 700, Japan

KENICHI MORI AND SETSUO KASHINO*

Faculty of Science, Okayama University, Tsushima, Okayama 700, Japan

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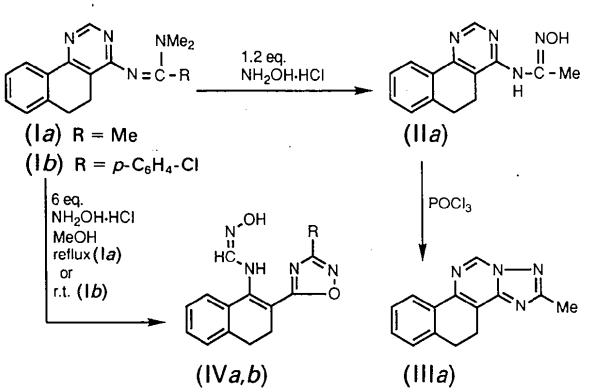
Abstract

X-ray structure analyses of the ring cleavage and recyclization products of N^1,N^1-dimethyl-N^2-(5,6-dihydrobenzo[*h*]quinazolin-4-yl)acetamidine (I*a*) and *p*-chloro-N^1,N^1-dimethyl-N^2-(5,6-dihydrobenzo[*h*]-quinazolin-4-yl)benzamidine (I*b*) revealed the structures of 2-(3-methyl[1,2,4]oxadiazol-5-yl)-3,4-dihydro-1-naphthylaminoformaldehyde oxime (IV*a*),

$C_{14}H_{14}N_4O_2$, and 2-(3-*p*-chlorophenyl[1,2,4]oxadiazol-5-yl)-3,4-dihydro-1-naphthylaminoformaldehyde oxime (IV*b*), $C_{19}H_{15}ClN_4O_2$. The different substituents had no significant effect on the conformations of the common moieties of both molecules. The N—OH groups, related by a center of symmetry, are linked by O—H···N hydrogen bonds: O···N 2.861 (2) for (IV*a*) and 2.809 (6) Å for (IV*b*).

Comment

We have reported the syntheses of the 5,6-dihydrobenzo[*h*][1,2,4]triazolo[1,5-*c*]quinazolines (III), containing alkyl substituents at the 2 position, *via* amidine (I) and oxime (II) derivatives from 4-amino-5,6-dihydrobenzo[*h*]quinazoline (Hirota, Sasaki, Yamamoto & Nakayama, 1991). The reaction of (I*a*) to form (II*a*) was carried out using 1.2 equivalents of hydroxylamine hydrochloride at room temperature. The analogous reaction of (I*b*) required 6 equivalents of hydroxylamine hydrochloride to complete the reaction at room temperature, and gave an abnormal product, (IV*b*). The reaction of (I*a*) with 6 equivalents of hydroxylamine hydrochloride at room temperature afforded the normal oxime (II*a*), but the abnormal oxime (IV*a*) was obtained when refluxing methanol conditions were used. This type of compound has not been reported previously.



The bond lengths and angles of the corresponding moieties of (IV*a*) and (IV*b*) are in agreement within experimental error. In (IV*a*) and (IV*b*), the 3,4-dihydrobenzene rings adopt pseudo C_2 conformations (Kitaigorodsky, 1973). The planar oxadiazole rings are linked to the 3,4-dihydronaphthyl groups through C(2)—C(11) bonds. The N(2)—C(11) bonds, which have double-bond character, each adopt a *cis* conformation with respect to the C(1)—C(2) double bond. The amidoxime side chains extend with similar conformations. The *p*-chlorophenyl ring of (IV*b*) is nearly coplanar with the oxadiazole ring.

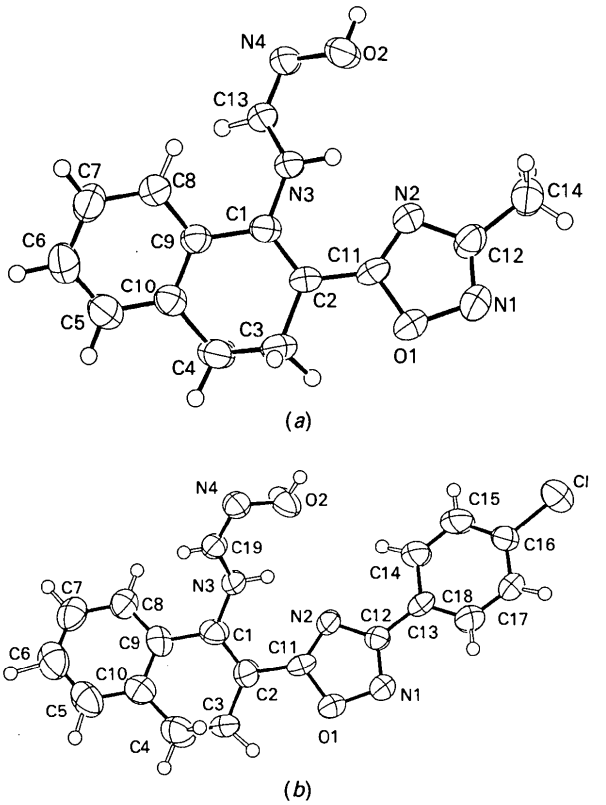


Fig. 1. Displacement ellipsoids with atomic numbering for (*a*) compound (IV*a*) and (*b*) compound (IV*b*). Ellipsoids of 50% probability are drawn for the non-H atoms; the H atoms are represented as spheres equivalent to $B = 1.0$ Å^2.

Experimental

Crystals of compound (IV*a*) were grown by slow evaporation from an ethanol solution, and those of compound (IV*b*) were grown from a methanol solution.

Compound (IV*a*)

Crystal data

$C_{14}H_{14}N_4O_2$	Mo $K\alpha$ radiation
$M_r = 270.29$	$\lambda = 0.7107$ Å
Monoclinic	Cell parameters from 25 reflections
$P2_1/a$	$\theta = 11.0$–$11.5°$
$a = 8.246$ (6) Å	$\mu = 0.088$ mm^{-1}
$b = 11.016$ (3) Å	$T = 295$ K
$c = 14.740$ (4) Å	Prism
$\beta = 96.84$ (4)°	0.50 × 0.20 × 0.10 mm
$V = 1329$ (1) Å^3	Colorless
$Z = 4$	
$D_x = 1.350$ Mg m^{-3}	

Data collection

Rigaku AFC-5*R* diffractometer	$R_{int} = 0.022$
$2\theta/\omega$ scans	$\theta_{max} = 25.0°$
Absorption correction: none	$h = 0 \rightarrow 9$
	$k = 0 \rightarrow 13$
	$l = -17 \rightarrow 17$

2654 measured reflections
2474 independent reflections
1649 observed reflections
$[I > 2.0\sigma(I)]$
3 standard reflections
monitored every 97
reflections
intensity variation: 2.7%

Refinement

Refinement on F
$R = 0.041$
$wR = 0.031$
$S = 1.43$
1649 reflections
238 parameters
All H-atom parameters
refined
$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{max} = 0.33$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Extinction correction:
secondary
Extinction coefficient:
1.66×10^{-6}
Atomic scattering factors
from *International Tables*
for X-ray Crystallography
(1974, Vol. IV)

Compound (IV*b*)

Crystal data

$C_{19}H_{15}ClN_4O_2$
$M_r = 366.81$
Monoclinic
$P2_1/n$
$a = 11.661$ (2) Å
$b = 9.410$ (2) Å
$c = 16.224$ (3) Å
$\beta = 103.42$ (1)°
$V = 1732$ (1) Å^3
$Z = 4$
$D_x = 1.407$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25
reflections
$\theta = 11.0$–$11.5°$
$\mu = 0.238$ mm^{-1}
$T = 298$ K
Prism
$0.38 \times 0.25 \times 0.15$ mm
Light yellow

Data collection

Rigaku AFC-5*R* diffractometer
$2\theta/\omega$ scans
Absorption correction:
none
3234 measured reflections
3074 independent reflections
1366 observed reflections
$[I > 3.0\sigma(I)]$
$R_{int} = 0.031$
$\theta_{max} = 25.0°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 11$
$l = -18 \rightarrow 18$
3 standard reflections
monitored every 97
reflections
intensity variation: 0.6%

Refinement

Refinement on F
$R = 0.057$
$wR = 0.037$
$S = 1.50$
1366 reflections
295 parameters
All H-atom parameters
refined
$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{max} = 0.22$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Atomic scattering factors
from *International Tables*
for X-ray Crystallography
(1974, Vol. IV)

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

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Compound (IV*a*)				
O(1)	0.1902 (2)	0.2155 (1)	0.56525 (9)	4.88 (8)
O(2)	−0.0575 (2)	0.0312 (1)	0.8924 (1)	5.09 (8)
N(1)	0.2078 (2)	0.0913 (2)	0.5409 (1)	5.3 (1)
N(2)	0.0881 (2)	0.1051 (1)	0.6697 (1)	3.79 (9)
N(3)	−0.0059 (2)	0.2335 (2)	0.8141 (1)	3.67 (9)
N(4)	−0.0010 (2)	0.1262 (1)	0.9528 (1)	4.20 (9)
C(1)	0.0293 (2)	0.3377 (2)	0.7658 (1)	3.3 (1)
C(2)	0.0897 (2)	0.3301 (2)	0.6842 (1)	3.5 (1)
C(3)	0.1264 (3)	0.4448 (2)	0.6343 (2)	4.3 (1)
C(4)	0.1800 (3)	0.5458 (2)	0.7008 (2)	4.4 (1)
C(5)	0.0170 (3)	0.6741 (2)	0.7991 (2)	4.4 (1)
C(6)	−0.0967 (3)	0.6863 (2)	0.8601 (2)	4.8 (1)
C(7)	−0.1699 (3)	0.5853 (2)	0.8910 (2)	4.8 (1)
C(8)	−0.1291 (3)	0.4710 (2)	0.8618 (1)	4.0 (1)
C(9)	−0.0119 (2)	0.4572 (2)	0.8017 (1)	3.3 (1)
C(10)	0.0601 (2)	0.5609 (2)	0.7689 (1)	3.6 (1)
C(11)	0.1189 (2)	0.2153 (2)	0.6433 (1)	3.6 (1)
C(12)	0.1471 (3)	0.0332 (2)	0.6054 (1)	4.1 (1)
C(13)	0.0183 (3)	0.2227 (2)	0.9072 (1)	3.9 (1)
C(14)	0.1441 (5)	−0.1018 (2)	0.6098 (2)	5.6 (2)
Compound (IV*b*)				
Cl	0.2298 (1)	0.0965 (2)	0.5183 (1)	6.59 (9)
O(1)	−0.0513 (3)	−0.2778 (4)	0.8679 (2)	4.5 (2)
O(2)	0.3603 (3)	0.0255 (5)	0.9687 (3)	6.3 (2)
N(1)	−0.0307 (4)	−0.2555 (5)	0.7866 (3)	4.7 (2)
N(2)	0.0826 (3)	−0.1094 (4)	0.8807 (3)	3.8 (2)
N(3)	0.2046 (4)	−0.0625 (5)	1.0428 (3)	3.8 (2)
N(4)	0.4062 (4)	−0.0306 (5)	1.0498 (3)	4.4 (2)
C(1)	0.1165 (5)	−0.1383 (6)	1.0691 (3)	4.3 (3)
C(2)	0.0237 (5)	−0.1977 (6)	1.0105 (4)	4.5 (3)
C(3)	−0.0674 (6)	−0.2780 (8)	1.0406 (4)	4.9 (3)
C(4)	−0.0105 (6)	−0.3646 (7)	1.1194 (5)	6.2 (4)
C(5)	0.0650 (6)	−0.2851 (9)	1.2711 (5)	6.7 (4)
C(6)	0.1287 (6)	−0.192 (1)	1.3294 (5)	6.9 (4)
C(7)	0.1847 (5)	−0.0807 (9)	1.3026 (4)	5.7 (4)
C(8)	0.1816 (5)	−0.0606 (7)	1.2186 (4)	4.6 (3)
C(9)	0.1203 (5)	−0.1543 (7)	1.1581 (3)	4.2 (3)
C(10)	0.0618 (5)	−0.2681 (7)	1.1853 (4)	4.7 (3)
C(11)	0.0202 (4)	−0.1865 (6)	0.9211 (3)	3.8 (3)
C(12)	0.0487 (4)	−0.1558 (6)	0.7994 (3)	3.5 (3)
C(13)	0.0947 (4)	−0.0979 (6)	0.7310 (3)	3.6 (3)
C(14)	0.1641 (6)	0.0210 (7)	0.7435 (4)	5.8 (4)
C(15)	0.2053 (6)	0.0791 (8)	0.6789 (5)	6.6 (4)
C(16)	0.1778 (5)	0.0215 (7)	0.6004 (4)	4.6 (3)
C(17)	0.1115 (6)	−0.0980 (8)	0.5859 (4)	6.0 (4)
C(18)	0.0702 (6)	−0.1553 (8)	0.6513 (4)	6.0 (4)
C(19)	0.3228 (5)	−0.0714 (6)	1.0817 (4)	3.8 (3)

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

Compound (IV*a*)			
O(1)—N(1)	1.427 (2)	C(2)—C(3)	1.511 (3)
O(1)—C(11)	1.353 (2)	C(2)—C(11)	1.433 (3)
O(2)—N(4)	1.417 (2)	C(3)—C(4)	1.514 (3)
N(1)—C(12)	1.295 (2)	C(4)—C(10)	1.501 (3)
N(2)—C(11)	1.308 (2)	C(5)—C(6)	1.381 (3)
N(2)—C(12)	1.368 (2)	C(5)—C(10)	1.384 (3)
N(3)—C(1)	1.399 (2)	C(6)—C(7)	1.369 (3)
N(3)—C(13)	1.369 (2)	C(7)—C(8)	1.385 (3)
N(4)—C(13)	1.278 (2)	C(8)—C(9)	1.395 (3)
C(1)—C(2)	1.358 (2)	C(9)—C(10)	1.399 (3)
C(1)—C(9)	1.474 (2)	C(12)—C(14)	1.489 (3)
N(1)—O(1)—C(11)	106.3 (2)	C(6)—C(7)—C(8)	120.2 (2)
O(1)—N(1)—C(12)	103.3 (2)	C(7)—C(8)—C(9)	120.6 (2)
C(11)—N(2)—C(12)	103.5 (2)	C(1)—C(9)—C(8)	122.1 (2)
C(1)—N(3)—C(13)	124.5 (2)	C(1)—C(9)—C(10)	118.7 (2)
O(2)—N(4)—C(13)	109.7 (2)	C(8)—C(9)—C(10)	119.0 (2)
N(3)—C(1)—C(2)	121.3 (2)	C(4)—C(10)—C(5)	122.0 (2)
N(3)—C(1)—C(9)	118.7 (2)	C(4)—C(10)—C(9)	118.8 (2)
C(2)—C(1)—C(9)	119.9 (2)	C(5)—C(10)—C(9)	119.3 (2)
C(1)—C(2)—C(3)	119.7 (2)	O(1)—C(11)—N(2)	111.9 (2)
C(1)—C(2)—C(11)	121.7 (2)	O(1)—C(11)—C(2)	117.9 (2)
C(3)—C(2)—C(11)	118.6 (2)	N(2)—C(11)—C(2)	130.2 (2)
C(2)—C(3)—C(4)	111.1 (2)	N(1)—C(12)—N(2)	115.0 (2)
C(3)—C(4)—C(10)	110.5 (2)	N(1)—C(12)—C(14)	122.4 (2)
C(6)—C(5)—C(10)	121.1 (2)	N(2)—C(12)—C(14)	122.6 (2)
C(5)—C(6)—C(7)	119.8 (2)	N(3)—C(13)—N(4)	125.9 (2)

O(1)—N(1)—C(12)—N(2)	1.1 (2)
O(1)—C(11)—N(2)—C(12)	1.4 (2)
N(1)—O(1)—C(11)—N(2)	−0.8 (2)
N(1)—C(12)—N(2)—C(11)	−1.6 (2)
C(11)—O(1)—N(1)—C(12)	−0.2 (2)
N(3)—C(1)—C(9)—C(8)	−21.7 (3)
C(1)—C(2)—C(3)—C(4)	32.0 (3)
C(1)—C(9)—C(10)—C(4)	−1.8 (3)
C(2)—C(3)—C(4)—C(10)	−51.4 (3)
C(3)—C(2)—C(1)—C(9)	4.7 (3)
C(3)—C(4)—C(10)—C(9)	37.9 (3)
N(2)—C(11)—C(2)—C(3)	−175.3 (2)
O(2)—N(4)—C(13)—N(3)	−2.0 (3)
N(4)—C(13)—N(3)—C(1)	−174.4 (2)
C(9)—C(1)—N(3)—C(13)	−41.4 (3)

Compound (IV*b*)

O(1)—N(1)	1.410 (5)	C(4)—C(10)	1.503 (8)
O(1)—C(11)	1.358 (5)	C(5)—C(6)	1.371 (9)
O(2)—N(4)	1.404 (5)	C(5)—C(10)	1.392 (8)
N(1)—C(12)	1.300 (5)	C(6)—C(7)	1.361 (8)
N(2)—C(11)	1.306 (5)	C(7)—C(8)	1.368 (7)
N(2)—C(12)	1.358 (5)	C(8)—C(9)	1.388 (7)
N(3)—C(1)	1.397 (6)	C(9)—C(10)	1.396 (7)
N(3)—C(19)	1.378 (6)	C(12)—C(13)	1.448 (6)
N(4)—C(19)	1.263 (6)	C(13)—C(14)	1.368 (7)
C(1)—C(2)	1.382 (6)	C(13)—C(18)	1.370 (7)
C(1)—C(9)	1.443 (6)	C(14)—C(15)	1.365 (8)
C(2)—C(3)	1.477 (7)	C(15)—C(16)	1.353 (8)
C(2)—C(11)	1.445 (6)	C(16)—C(17)	1.354 (7)
C(3)—C(4)	1.530 (8)	C(17)—C(18)	1.372 (8)
N(1)—O(1)—C(11)	106.7 (4)	C(8)—C(9)—C(10)	118.2 (6)
O(1)—N(1)—C(12)	103.1 (4)	C(4)—C(10)—C(5)	121.6 (7)
C(11)—N(2)—C(12)	103.8 (4)	C(4)—C(10)—C(9)	118.2 (6)
C(1)—N(3)—C(19)	123.9 (5)	C(5)—C(10)—C(9)	120.1 (7)
O(2)—N(4)—C(19)	109.6 (5)	O(1)—C(11)—N(2)	111.4 (5)
N(3)—C(1)—C(2)	120.7 (5)	O(1)—C(11)—C(2)	117.5 (5)
N(3)—C(1)—C(9)	120.4 (5)	N(2)—C(11)—C(2)	130.9 (5)
C(2)—C(1)—C(9)	119.0 (5)	N(1)—C(12)—N(2)	115.0 (5)
C(1)—C(2)—C(3)	119.2 (5)	N(1)—C(12)—C(13)	121.7 (5)
C(1)—C(2)—C(11)	119.8 (5)	N(2)—C(12)—C(13)	123.2 (5)
C(3)—C(2)—C(11)	121.0 (5)	C(12)—C(13)—C(14)	120.4 (6)
C(2)—C(3)—C(4)	110.1 (6)	C(12)—C(13)—C(18)	122.9 (6)
C(3)—C(4)—C(10)	109.7 (6)	C(14)—C(13)—C(18)	116.7 (6)
C(6)—C(5)—C(10)	120.0 (8)	C(13)—C(14)—C(15)	121.0 (6)
C(5)—C(6)—C(7)	119.8 (7)	C(14)—C(15)—C(16)	120.9 (6)
C(6)—C(7)—C(8)	121.2 (7)	C(15)—C(16)—C(17)	119.8 (6)
C(7)—C(8)—C(9)	120.5 (7)	C(16)—C(17)—C(18)	118.8 (6)
C(1)—C(9)—C(8)	122.2 (6)	C(13)—C(18)—C(17)	122.7 (6)
C(1)—C(9)—C(10)	119.6 (6)	N(3)—C(19)—N(4)	125.2 (6)

O(1)—N(1)—C(12)—N(2)	−0.2 (6)
O(1)—C(11)—N(2)—C(12)	−0.4 (6)
N(1)—O(1)—C(11)—N(2)	0.3 (6)
N(1)—C(12)—N(2)—C(11)	0.4 (6)
C(11)—O(1)—N(1)—C(12)	−0.1 (5)
N(3)—C(1)—C(9)—C(8)	22.9 (8)
C(1)—C(2)—C(3)—C(4)	−38.1 (8)
C(1)—C(9)—C(10)—C(4)	−4.3 (8)
C(2)—C(3)—C(4)—C(10)	54.3 (8)
C(3)—C(2)—C(1)—C(9)	−1.2 (8)
C(3)—C(4)—C(10)—C(9)	−34.7 (8)
N(2)—C(11)—C(2)—C(3)	171.4 (6)
O(2)—N(4)—C(19)—N(3)	0.2 (8)
N(1)—C(12)—C(13)—C(14)	169.9 (5)
N(4)—C(19)—N(3)—C(1)	163.5 (5)
C(9)—C(1)—N(3)—C(19)	40.8 (7)

The structures were solved by direct methods using *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984) and refined by full-matrix least squares using *TEXSAN* (Molecular Structure Corporation, 1985). H atoms were found by difference synthesis and refined isotropically. The displacement ellipsoids were drawn with the aid of *ORTEP*II (Johnson, 1976). Most of the calculations were performed on a VAX 3100 computer using *TEXSAN* at the X-ray Laboratory of Okayama University.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71719 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1051]

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3-Sulfure de 3-Mercapto-5,7-diméthyl-2-phényl-3*H*-[1,4,2]diazaphospholo-[1,5-*a*]pyridinium

N. Rodier

Laboratoire de Chimie minérale, Faculté des Sciences pharmaceutiques et biologiques, 5 Rue J.-B. Clément, 92296 Châtenay-Malabry CEDEX, France

J.-M. Cense

Laboratoire d'Informatique appliquée à la Chimie, ENSCP, 11 Rue P. et M. Curie, 75231 Paris CEDEX 05, France

J.-M. Robert, S. Robert-Piessard, J. Courant et G. Le Baut

Laboratoire de Chimie thérapeutique, Faculté des Sciences pharmaceutiques, 1 Rue Gaston-Veil, 44035 Nantes CEDEX, France

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Abstract

The entire molecule of 5,7-dimethyl-2-phenyl-3-thioxo-3*H*-[1,4,2]diazaphospholo[1,5-*a*]pyridinium-3-thiolate, $C_{14}H_{13}N_2PS_2$, with the exception of the two